EXHIBIT E

QUALITY ASSURANCE/QUALITY CONTROL PROCEDURES AND REQUIREMENTS

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Exhibit E –- Quality Assurance/Quality Control Procedures and Requirements

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1.0 OVERVIEW

Quality Assurance (QA) and Quality Control (QC) are integral parts of the U.S. Environmental Protection Agency's (USEPA's) Contract Laboratory Program (CLP). The QA process consists of management review and oversight at the planning, implementation, and completion stages of the environmental data collection activity, and ensures that data provided are of the quality required. The QC process includes those activities required during data collection to produce the data quality desired and to document the quality of the collected data.

1.1 Quality Assurance/Quality Control (QA/QC) Activities

During the planning of an environmental data collection program, QA activities focus on defining data quality criteria and designing a QC system to measure the quality of data being generated. During the implementation of the data collection effort, QA activities ensure that the QC system is functioning effectively, and that the deficiencies uncovered by the QC system are corrected. After environmental data are collected, QA activities focus on assessing the quality of data obtained to determine its suitability to support enforcement or remedial decisions.

1.1.1

This exhibit describes the overall QA/QC operations and the processes by which the CLP meets the QA/QC objectives defined above. This contract requires a variety of QA/QC activities. These contract requirements are the minimum QC operations necessary to satisfy the analytical requirements associated with the determination of the different method analytes. These QC operations are designed to facilitate laboratory comparison by providing USEPA with comparable data from all Contractors. These requirements do not release the analytical Contractor from maintaining their own QC checks on method and instrument performance.

2.0 INTRODUCTION

Appropriate use of data generated under the large range of analytical conditions encountered in environmental analyses requires reliance on the Quality Control (QC) procedures and criteria incorporated into the methods. The methods in this contract have been validated on samples typical of those received by the laboratories in the Contract Laboratory Program (CLP). However, the validation of these methods does not guarantee that they perform equally well for all sample matrices encountered. Inaccuracies can also result from causes other than unanticipated matrix effects, such as sampling artifacts, equipment malfunctions, and operator error. Therefore, the QC component of each method is indispensable.

The data acquired from QC procedures are used to estimate and evaluate the information content of analytical results and to determine the necessity for or the effect of corrective action procedures. The parameters used to estimate information content include precision, accuracy, and other quantitative and qualitative indicators. In addition, QC procedures give an overview of the activities required in an integrated program to generate data of known and documented quality required to meet defined objectives.

2.1 Quality Assurance/Quality Control (QA/QC) Program Components

2.1.1 The necessary components of a complete QA/QC program include internal QC criteria that demonstrate acceptable levels of performance, as determined by QA review. External review of data and procedures is accomplished by the monitoring activities of the National Program Office, Regional Data Users, Sample Management Office (SMO), and the Quality Assurance Technical Support (QATS) Laboratory. Each external review accomplishes a different purpose. These reviews are described in specific sections of this exhibit. Laboratory evaluation samples, electronic data audits, and data packages provide an external QA reference for the program. A Contractor on-site evaluation system is also part of the external QA monitoring. A feedback loop provides the results of the various review functions to the Contractors through direct communications with the USEPA Regional CLP Project Officer (CLP PO).

2.1.2 This exhibit does not provide specific instructions for constructing QA Plans, QC systems, or a QA organization. It is, however, an explanation of the QA/QC requirements of the program. It outlines some minimum standards for QA/QC programs. It also includes specific items that are required in a Quality Assurance Plan (QAP) and by the QA/QC documentation detailed in this contract. Delivery of this documentation provides USEPA with a complete data package which will stand alone, and limits the need for contact with the Contractor or with an analyst, at a later date, if some aspect of the analysis is questioned.

2.1.3 In order to assure that the product delivered by the Contractor meets the requirements of the contract, and to improve interlaboratory data comparison, USEPA requires the following from the Contractor:

- Preparation of, and adherence to, a written QAP, the elements of which are designated in Section 5;
- Preparation of, and adherence to, Standard Operating Procedures (SOPs) as described in Section 6;

- Adherence to the analytical methods and associated QC requirements specified in the contract;

- Verification of analytical standards and documentation of the purity of neat materials and the purity and accuracy of solutions obtained from private chemical supply houses;

- Submission of all raw data and pertinent documentation for Regional review;

- Participation in the analysis of laboratory evaluation samples, including adherence to corrective action procedures;

- Submission, upon request, of instrument data tapes and applicable documentation for tape audits, including a copy of the Sample Data Package;

- Participation in on-site laboratory evaluations, including adherence to corrective action procedures; and

- Submission of all original documentation generated during sample analyses for USEPA review.

3.0 GENERAL QUALITY ASSURANCE/QUALITY CONTROL (QA/QC) PRACTICES

The Contractor shall adhere to good laboratory practices for laboratory cleanliness with regard to glassware and apparatus. The Contractor shall also adhere to good laboratory practices with regard to reagents, solvents, and gases. For additional guidelines regarding these general laboratory procedures, see the Handbook for Analytical Quality Control in Water and Wastewater Laboratories USEPA-600/4-79-019, USEPA Environmental Monitoring Systems Laboratory, Cincinnati, Ohio, September 1982.

4.0 SPECIFIC QUALITY ASSURANCE/QUALITY CONTROL (QA/QC) PROCEDURES

The QA/QC procedures defined herein shall be used by the Contractor when performing the methods specified in Exhibit D. When additional QA/QC procedures are specified in the methods in Exhibit D, the Contractor shall also follow these procedures.

NOTE: The cost of performing all QA/QC procedures specified in this Statement of Work (SOW) is included in the price of performing the bid lot.

4.1 Purpose

4.1.1 The purpose of this document is to provide a uniform set of procedures for the analysis of organic samples, documentation of methods and their performance, and verification of the sample data generated. The program will also assist laboratory personnel in recalling and defending their actions under cross examination if required to present court testimony in enforcement case litigation. Although it is impossible to address all analytical situations in one document, the approach taken here is to define the minimum requirements for all major steps relevant to any organic low concentration analysis.

4.1.2 The primary function of the QA/QC program is the definition of procedures for the evaluation and documentation of analytical methodologies and the reduction and reporting of data. The objective is to provide a uniform basis for sample handling, instrument and methods maintenance, performance evaluation, and analytical data gathering and reporting. In many instances where methodologies are available, specific QC procedures are incorporated into the method documentation (Exhibit D).

4.2 Laboratory Audit and Intercomparison Study Program

The Contractor is required to participate in the Laboratory Audit and Intercomparison Study Program run by USEPA. The Contractor can expect to analyze at least two Performance Evaluation (PE) samples per calendar quarter during the contract period for organic low concentration analyses.

4.3 Annual Verification of Method Detection Limits (MDLs)

The Contractor shall perform annual verification of MDLs in accordance with the specifications in Exhibit D. All the MDLs shall meet the Contract Required Quantitation Limits (CRQLs) specified in Exhibit C.

4.4 Quality Assurance/Quality Control Measurements

4.4.1 In this Exhibit, as well as other places within this SOW, the term "analytical sample" is used in discussing the required frequency or placement of certain QA/QC measurements. As the term is used, analytical sample includes all field samples, including PE samples, received from an external source. It also includes all required QA/QC samples [requested Matrix Spike/Matrix Spike Duplicate(s) (MS/MSD), and Laboratory Control Sample (LCS)] except those directly related to instrument calibration or calibration verification (calibration standards, Initial Calibration, Continuing Calibration, and tunes).

4.4.2 In order for the QA/QC information to reflect the status of the samples analyzed, all samples and their QA/QC analysis shall be analyzed under the same operating and procedural conditions.

4.4.3 If any QC measurement fails to meet contract criteria, the analytical measurement must not be repeated prior to taking the appropriate corrective action as specified in Exhibit D.

4.4.4 The Contractor shall report all QC data in the exact format specified in Exhibits B and H.

4.4.5 In addition, the Contractor shall establish a QA program with the objective of providing sound analytical chemical measurements. This program shall incorporate the QC procedures, any necessary corrective action, and all documentation required during data collection, as well as the quality assessment measures performed by management to ensure acceptable data production.

5.0 QUALITY ASSURANCE PLAN (QAP)

5.1 Introduction

The Contractor shall establish a Quality Assurance (QA) program with the objective of providing sound analytical chemical measurements. This program shall incorporate the Quality Control (QC) procedures, any necessary corrective action, all documentation required during data collection, and the quality assessment measures performed by management to ensure acceptable data production.

5.1.1 As evidence of such a program, the Contractor shall prepare a written QAP which describes the procedures that are implemented to achieve the following:

- Maintain data integrity, validity, and usability;
- Ensure that analytical measurement systems are maintained in an acceptable state of stability and reproducibility;
- Detect problems through data assessment and establish corrective action procedures which keep the analytical process reliable; and
- Document all aspects of the measurement process in order to provide data which are technically sound and legally defensible.

5.1.2 The QAP must present, in specific terms, the policies, organization, objectives, functional guidelines, and specific QA/QC activities designed to achieve the data quality requirements in this contract. Where applicable, Standard Operating Procedures (SOPs) pertaining to each element shall be included or referenced as part of the QAP. The QAP shall be paginated consecutively in ascending order. The QAP shall be available during on-site laboratory evaluations and shall be submitted within 7 days of written request by the USEPA Regional Contract Laboratory Program Project Officer (CLP PO). Additional information relevant to the preparation of a QAP can be found in USEPA and American Society for Testing and Materials (ASTM) publications.

5.2 Required Elements of a Quality Assurance Plan

The required elements of a laboratory's QAP are outlined in this section. This outline should be used as a framework for developing the QAP.

A. Organization and Personnel

1. QA Policy and Objectives

2. QA Management

a. Organization

b. Assignment of QA/QC Responsibilities

c. Reporting Relationships

d. QA Document Control Procedures

e. QA Program Assessment Procedures

3. Personnel

a. Resumes

b. Education and Experience Pertinent to this Contract

c. Training Progress

B. Facilities and Equipment

1. Instrumentation and Backup Alternatives

2. Maintenance Activities and Schedules

C. Document Control

1. Laboratory Notebook Policy

2. Sample Tracking/Custody Procedures

3. Logbook Maintenance and Archiving Procedures

4. Sample Delivery Group (SDG) File Organization, Preparation, and Review Procedures

5. Procedures for Preparation, Approval, Review, Revision, and Distribution of Standard Operating Procedures (SOPs)

6. Process for Revision of Technical or Documentation Procedures

D. Analytical Methodology

1. Calibration Procedures and Frequency

2. Sample Preparation Procedures

3. Sample Analysis Procedures

4. Standards Preparation Procedures

5. Decision Processes, Procedures, and Responsibility for Initiation of Corrective Action

E. Data Generation

1. Data Collection Procedures

2. Data Reduction Procedures

3. Data Validation Procedures

4. Data Reporting and Authorization Procedures

F. Quality Assurance

1. Data Quality Assurance

2. Systems/Internal Audits

3. Performance/External Audits

4. Corrective Action Procedures

5. QA Reporting Procedures

6. Responsibility Designation

G. Quality Control

1. Solvent, Reagent, and Adsorbent Check Analysis

2. Reference Material Analysis

3. Internal QC Checks

4. Corrective Action and Determination of QC Limit Procedures

5. Responsibility Designation

5.3 Updating and Submitting the Quality Assurance Plan

5.3.1 Initial Submission. During the contract solicitation process, the Contractor is required to submit its QAP to the CLP Contracting Officer. Within sixty days after contract award, the Contractor shall maintain on file a revised QAP, fully compliant with the requirements of this contract. The revised QAP will become the official QAP under the contract and may be used during legal proceedings. The Contractor shall maintain the QAP on file at the Contractor's facility for the term of the contract. Both the initial submission and the revised QAP shall be paginated consecutively in ascending order. The revised QAP shall include:

- Changes resulting from (1) the Contractor's internal review of their organization, personnel, facility, equipment, policy and procedures and (2) the Contractor's implementation of the requirements of the contract; and

- Changes resulting from USEPA's review of the laboratory evaluation sample data, bidder supplied documentation, and recommendations made during the pre-award on-site laboratory evaluation.

5.3.1.1 The Contractor shall send a copy of the latest version of the QAP within 7 days of a request from a USEPA Regional CLP PO. The USEPA requestor will designate the recipients.

5.3.2 Subsequent Updates and Submissions. During the term of contract, the Contractor shall amend the QAP when the following circumstances occur:

- USEPA modifies the contract;
- USEPA notifies the Contractor of deficiencies in the QAP document;
- USEPA notifies the Contractor of deficiencies resulting from USEPA's review of the Contractor's performance;
- The Contractor identifies deficiencies resulting from their internal review of their QAP document;
- The Contractor's organization, personnel, facility, equipment, policy, or procedures change; or
- The Contractor identifies deficiencies resulting from the internal review of their organization, personnel, facility, equipment, policy, or procedures changes.

5.3.2.1 The Contractor shall amend the QAP within 30 days of when the circumstances listed above result in a discrepancy between what was previously described in the QAP and what is presently occurring at the Contractor's facility. When the QAP is amended, all changes in the QAP shall be clearly marked (e.g., a bar in the margin indicating where the change is found in the document, or highlighting the change by underlining the change, bold printing the change, or using a different print font). The amended section pages shall have the date on which the changes were implemented. The Contractor shall incorporate all amendments to the latest version of the QAP document. The Contractor shall archive all amendments to the QAP document for future reference by USEPA.

5.3.2.2 The Contractor shall send a copy of the latest version of the QAP document within 7 days of a written request by the Regional CLP PO as directed. The USEPA requestor will designate the recipients.

5.4 Corrective Actions

If the Contractor fails to adhere to the requirements listed in this section, the Contractor may expect, but USEPA is not limited to the following actions: reduction in the numbers of samples sent under this contract, suspension of sample shipment to the Contractor, data package audit, electronic data audit (i.e., Gas Chromatograph/Mass Spectrometer (GC/MS) tape audit), an on-site laboratory evaluation, remedial performance evaluation sample, and/or contract sanctions.

6.0 STANDARD OPERATING PROCEDURES (SOPs)

6.1 Introduction

In order to obtain reliable results, adherence to prescribed analytical methodology is imperative. In any operation that is performed on a repetitive basis, reproducibility is best accomplished through the use of Standard Operating Procedures (SOPs). As defined by USEPA, a SOP is a written document which provides directions for the step-by-step execution of an operation, analysis, or action which is commonly accepted as the method for performing certain routine or repetitive tasks.

6.1.1 SOPs prepared by the Contractor shall be functional (i.e., clear, comprehensive, up-to-date, and sufficiently detailed to permit duplication of results by qualified analysts). The SOPs shall be paginated consecutively in ascending order.

6.1.2 All SOPs shall reflect activities as they are currently performed in the laboratory. In addition, all SOPs shall be:

- Consistent with current USEPA regulations, guidelines, and the Contract Laboratory Program (CLP) contract’s requirements.
- Consistent with instrument(s) manufacturer’s specific instruction manuals.
- Available to USEPA during an on-site laboratory evaluation. A complete set of SOPs shall be bound together and available for inspection at such evaluations. During on-site laboratory evaluations, laboratory personnel may be asked to demonstrate the application of the SOPs.
- Available to the designated recipients within 7 days, upon request by the USEPA Regional CLP Project Officer (CLP PO).
- Capable of providing for the development of documentation that is sufficiently complete to record the performance of all tasks required by the protocol.
- Capable of demonstrating the validity of data reported by the Contractor and explain the cause of missing or inconsistent results.
- Capable of describing the corrective measures and feedback mechanism utilized when analytical results do not meet protocol requirements.
- Reviewed regularly and updated as necessary when contract, facility, or Contractor procedural modifications are made.
- Archived for future reference in usability or evidentiary situations.
- Available at specific work stations as appropriate.
- Subject to a document control procedure which precludes the use of outdated or inappropriate SOPs.

6.2 Format

The format for SOPs may vary depending upon the type of activity for which they are prepared; however, at a minimum, the following sections shall be included:

- Title page;
- Scope and application;
- Definitions;
- Procedures;
- Quality Control (QC) limits;
- Corrective action procedures, including procedures for secondary review of information being generated;
- Documentation description and example forms;
- Miscellaneous notes and precautions; and
- References.

6.3 Requirements

The Contractor shall maintain the following SOPs:

6.3.1 Evidentiary SOPs for required Chain-of-Custody and document control are discussed in Exhibit F.

6.3.2 Sample Receipt and Storage

- Sample receipt and identification logbooks;
- Refrigerator temperature logbooks; and
- Security precautions.

6.3.3 Sample Preparation

- Reagent purity check procedures and documentation;
- Extraction procedures;
- Extraction bench sheets; and
- Extraction logbook maintenance.

6.3.4 Glassware Cleaning

6.3.5 Calibration (Balances, etc.)

- Procedures;
- Frequency requirements;
- Preventive maintenance schedule and procedures; and
- Acceptance criteria and corrective actions.

6.3.6 Analytical Procedures (for each analytical system)

- Instrument performance specifications;
- Instrument operating procedures;
- Data acquisition system operation;
- Procedures when automatic quantitation algorithms are overridden;
- QC required parameters;
- Analytical run/injection logbooks; and
- Instrument error and editing flag descriptions and resulting corrective actions.

6.3.7 Maintenance Activities (for each analytical system)

- Preventative maintenance schedule and procedures;
- Corrective maintenance determinants and procedures; and
- Maintenance authorization.

6.3.8 Analytical Standards

- Standard coding/identification and inventory system;
- Standards preparation logbook(s);
- Standard preparation procedures;
- Procedures for equivalency/traceability analyses and documentation;
- Purity logbook (primary standards and solvents);
- Storage, replacement, and labeling requirements; and
- QC and corrective action measures.

6.3.9 Data Reduction Procedures

- Data processing systems operation;
- Outlier identification methods;
- Identification of data requiring corrective action; and
- Procedures for format and/or forms for each operation.

6.3.10 Documentation Policy/Procedures

- Contractor/analyst's notebook policy, including review policy;
- Complete SDG File contents;
- Complete SDG File organization and assembly procedures, including review policy; and
- Document inventory procedures, including review policy.

6.3.11 Data Validation/Self-Inspection Procedures

- Data flow and chain-of-command for data review;
- Procedures for measuring precision and accuracy;
- Evaluation parameters for identifying systematic errors;
- Procedures to assure that hardcopy and electronic deliverables are complete and compliant with the requirements in the Statement of Work (SOW) Exhibits B and H;
- Procedures to assure that hardcopy deliverables are in agreement with their comparable electronic deliverables;
- Demonstration of internal QA inspection procedure (demonstrated by supervisory sign-off on personal notebooks, internal laboratory evaluation samples, etc.);
- Frequency and type of internal audits (e.g., random, quarterly, spot checks, perceived trouble areas);
- Demonstration of problem identification, corrective actions, and resumption of analytical processing. Sequence resulting from internal audit (i.e., QA feedback); and
- Documentation of audit reports (internal and external), response, corrective action, etc.

6.3.12 Data Management and Handling

- Procedures for controlling and estimating data entry errors;
- Procedures for reviewing changes to data and deliverables and ensuring traceability of updates;
- Lifecycle management procedures for testing, modifying, and implementing changes to existing computing systems including hardware, software, and documentation or installing new systems;
- Database security, backup, and archival procedures including recovery from system failures;
- System maintenance procedures and response time;
- Individual(s) responsible for system operation, maintenance, data integrity, and security;
- Specifications for staff training procedures; and
- Virus protection procedures for software and electronic deliverables.

6.4 Updating and Submitting SOPs

6.4.1 Initial Submission. During the contract solicitation process, the Contractor is required to submit their SOPs to the CLP Contracting Officer. Within 60 days after contract award, the Contractor shall maintain on file a complete revised set of SOPs, fully compliant with the requirements of this contract. The revised SOPs will become the official SOPs under the contract and may be used during legal

proceedings. The Contractor shall maintain the complete set of SOPs on file at the Contractor's facility for the term of the contract. Both the initial submission of SOPs and the revised SOPs shall be paginated consecutively in ascending order. The revised SOPs shall include:

- Changes resulting from 1) the Contractor's internal review of their procedures, and 2) the Contractor's implementation of the requirements of the contract, and
- Changes resulting from USEPA's review of the laboratory evaluation sample data, bidder supplied documentation, and recommendations made during the preaward on-site laboratory evaluation.

6.4.1.1 The Contractor shall send a complete set of the latest version of SOPs or individually requested SOPs within 7 days of a request from an USEPA Regional CLP PO. The USEPA requestor will designate the recipients.

6.4.2 Subsequent Updates and Submissions. During the term of the contract, the Contractor shall amend the SOPs when the following circumstances occur:

- USEPA modifies the contract;
- USEPA notifies the Contractor of deficiencies in their SOP's documentation;
- USEPA notifies the Contractor of deficiencies resulting from USEPA's review of the Contractor's performance;
- The Contractor's procedures change;
- The Contractor identifies deficiencies resulting from the internal review of their SOPs documentation; or
- The Contractor identifies deficiencies resulting from the internal review of their procedures.

6.4.2.1 Existing SOPs shall be amended or new SOPs shall be written within 30 days of when the circumstances listed above result in a discrepancy between what was previously described in the SOPs and what is presently occurring at the Contractor's facility. All changes in the SOPs shall be clearly marked (e.g., a bar in the margin indicating where the change is in the document, or highlighting the change by underlining the change, bold printing the change, or using a different print font). The amended/new SOPs shall have the date on which the changes were implemented.

6.4.2.2 When existing SOPs are amended or new SOPs are written, the Contractor shall document the reasons for the changes and maintain the amended SOPs or new SOPs on file. Documentation of the reasons for the changes shall be maintained on file with the amended SOPs or new SOPs.

6.4.2.3 Documentation of the reason(s) for changes to the SOPs shall also be submitted along with the SOPs. An alternate delivery schedule for submitting the letter and amended/new SOPs may be proposed by the Contractor, but it is the sole decision of the USEPA Contracting Officer to approve or disapprove the alternate delivery schedule. If an alternate delivery schedule is proposed,

the Contractor shall describe in a letter to the USEPA Regional CLP PO and the Contracting Officer why it is unable to meet the delivery schedule listed in this section. The USEPA Regional CLP PO may grant an extension of up to 30 days for amending/writing new SOPs. An extension for amending/writing new SOPs beyond 30 days must be approved by the USEPA Contracting Officer. Similarly, an extension of up to 14 days for submission of the letter documenting the reasons for the changes and for submitting amended/new SOPs may be approved by the USEPA Regional CLP PO. An extension beyond the 14 days must be approved by the USEPA Contracting Officer. The Contractor shall proceed and not assume that an extension will be granted until so notified by the USEPA Regional CLP PO and/or Contracting Officer.

6.5 Corrective Action

If the Contractor fails to adhere to the requirements listed in this section, the Contractor may expect, but USEPA is not limited to the following actions: reduction in the number of samples sent under this contract, suspension of sample shipment to the Contractor, data package audit, electronic data audit, an on-site laboratory evaluation, remedial performance evaluation sample, and/or contract sanctions.

7.0 CONTRACT COMPLIANCE SCREENING (CCS)

7.1 Overview

7.1.1 CCS is one aspect of the Government's contractual right of inspection of analytical data. CCS examines the Contractor's adherence to the contract requirements based on the Sample Data Package delivered to USEPA.

7.1.2 CCS is performed by the Sample Management Office (SMO) under the direction of USEPA. To assure a uniform review, a set of standardized procedures has been developed to evaluate the Sample Data Package submitted by a Contractor against the technical and completeness requirements of the contract. USEPA reserves the right to add and/or delete individual checks.

7.2 CCS Results

CCS results are mailed to the Contractor and all other data recipients. The Contractor has a period of time to correct deficiencies. The Contractor shall send all corrections to the Regional client and SMO within 6 business days. CCS results are used in conjunction with other information to measure overall Contractor performance and to take appropriate actions to correct deficiencies in performance.

7.3 CCS Trend Report

USEPA may generate a CCS trend report which summarizes CCS results over a given period of time. USEPA may send the CCS trend report or discuss the CCS trend report during an on-site laboratory evaluation. In a detailed letter to the USEPA Regional Contract Laboratory Program Project Officer (CLP PO) and Contracting Officer, the Contractor shall address the deficiencies and the subsequent corrective action implemented by the Contractor to correct the deficiencies within 14 days of receipt of the report of the on-site laboratory evaluation. An alternate delivery schedule may be proposed by the Contractor, but it is the sole decision of USEPA to approve or disapprove the alternate delivery schedule. If an alternate delivery schedule is proposed, the Contractor shall describe in a letter to the USEPA Regional CLP PO and Contracting Officer why it is unable to meet the delivery schedule listed in this section. The USEPA Regional CLP PO may grant an extension of up to 14 days for the Contractor's response to the CCS trend report. An extension beyond 14 days must be approved by the USEPA Contracting Officer. The Contractor shall proceed and not assume that an extension will be granted until so notified by the appropriate USEPA official.

7.4 Corrective Actions

7.4.1 If new Standard Operating Procedures (SOPs) are required to be written, or if existing SOPs are required to be rewritten or amended because of deficiencies and subsequent corrective action implemented by the Contractor, the Contractor shall write/amend the SOPs per the requirements listed in Section 6.

7.4.2 If the Contractor fails to adhere to the requirements listed in this section, the Contractor may expect, but USEPA is not limited to the following actions: reduction in the number of samples sent under the contract, suspension of sample shipment to the Contractor, data package audit, electronic data audit, an on-site laboratory evaluation, a remedial performance evaluation sample, and/or contract sanctions.

8.0 ANALYTICAL STANDARDS REQUIREMENTS

8.1 Overview

USEPA may not supply analytical reference standards either for direct analytical measurements or for the purpose of traceability. All contract laboratories shall be required to prepare from materials or purchase from private chemical supply houses those standards necessary to successfully and accurately perform the analyses required in this protocol.

8.2 Preparation of Chemical Standards from the Neat High Purity Bulk Material

8.2.1 If the laboratory cannot obtain analytical reference standards, the laboratory may prepare its own chemical standards. Laboratories shall obtain the highest purity possible when purchasing chemical standards; standards purchased at less than 97% purity shall be documented as to why a higher purity could not be obtained.

8.2.2 If required by the manufacturer, the chemical standards shall be kept refrigerated when not being used in the preparation of standard solutions. Proper storage of chemicals is essential in order to safeguard them from decomposition.

8.2.3 The purity of a compound can sometimes be misrepresented by a chemical supply house. Since knowledge of purity is needed to calculate the concentration of solute in a solution standard, it is the Contractor's responsibility to have analytical documentation ascertaining that the purity of each compound is correctly stated. Purity confirmation, when performed, should use appropriate techniques. Use of two or more independent methods is recommended. The correction factor for impurity when weighing neat materials in the preparation of solution standards is:

EQ. 1

$$\text{weight of impure compound} = \frac{\text{weight of pure compound}}{(\text{percent purity}/100)}$$

Where: "weight of pure compound" is that required to prepare a specific volume of a solution standard of a specified concentration.

8.2.4 When compound purity is assayed to be 97 percent or greater, the weight may be used without correction to calculate the concentration of the stock standard. If the compound purity is assayed to be less than 97 percent, the weight shall be corrected when calculating the concentration of the stock solution.

8.2.5 Mis-identification of compounds occasionally occurs and it is possible that a mislabeled compound may be received from a chemical supply house. It is the Contractor's responsibility to have analytical documentation ascertaining that all compounds used in the preparation of solution standards are correctly identified. Identification confirmation, when performed, shall use Gas Chromatography/Mass Spectrometry (GC/MS) analysis on at least two different analytical columns, or other appropriate techniques.

8.2.6 Calculate the weight of material to be weighed out for a specified volume, taking into account the purity of the compound and the

desired concentration. A second person shall verify the accuracy of the calculations. Check balances for accuracy with a set of standard weights every 12 hours. All weighing shall be performed on an analytical balance to the nearest 0.1 milligram (mg) and verified by a second person. The solvent used to dissolve the solute shall be compatible with the protocol in which the standard is to be used; the solute shall be soluble, stable, and non-reactive with the solvent. In the case of a multicomponent solution, the components must not react with each other.

8.2.7 Transfer the solute to a volumetric flask and dilute to the specified solution volume with solvent after ensuring dissolution of the solute in the solvent. Sonication or warming may be performed to promote dissolution of the solute. This solution shall be called the primary standard and all subsequent dilutions shall be traceable back to the primary standard.

8.2.8 Log notebooks are to be kept for all weighing and dilutions. All subsequent dilutions from the primary standard and the calculations for determining their concentrations are to be recorded and verified by a second person. All solution standards are to be refrigerated, if required, when not in use. All solution standards are to be clearly labeled as to the identity of the analyte or analytes, concentration, date prepared, solvent, and initials of the preparer.

8.3 Purchase of Chemical Standards Already in Solution

Solutions of analytical reference standards can be purchased by Contractors provided they meet the following criteria.

8.3.1 Contractors shall maintain documentation of the purity confirmation of the material to verify the integrity of the standard solutions they purchase.

8.3.2 The Contractor shall purchase standards for which the quality is demonstrated statistically and analytically by a method of the supplier's choice. One way this can be demonstrated is to prepare and analyze three solutions: a high standard, a low standard, and a standard at the target concentration (Sections 8.3.2.1 and 8.3.2.2). The supplier must then demonstrate that the analytical results for the high standard and low standard are consistent with the difference in theoretical concentrations. This is done by the student's t-test in Section 8.3.2.4. If this is achieved, the supplier must then demonstrate that the concentration of the target standard lies midway between the concentrations of the low and high standards. This is done by the student's t-test in Section 8.3.2.5. Thus the standard is certified to be within 10 percent of the target concentration using the equations in Section 8.3.2.6. If the procedure described above is used, the supplier must document that the following have been achieved.

8.3.2.1 Two solutions of identical concentration shall be prepared independently from neat materials. An aliquot of the first solution shall be diluted to the intended concentration (the "target standard"). One aliquot is taken from the second solution and diluted to a concentration 10 percent greater than the target standard. This is called the "high standard". One further aliquot is taken from the second solution and diluted to a concentration 10 percent less than the target standard. This is called the "low standard".

8.3.2.2 Six replicate analyses of each standard (a total of 18 analyses) shall be performed in the following sequence: low standard, target standard, high standard; low standard, target standard, high standard; etc.

8.3.2.3 The mean and variance of the six results for each solution shall be calculated:

EQ. 2

$$\text{MEAN} = \frac{Y_1 + Y_2 + Y_3 + Y_4 + Y_5 + Y_6}{6}$$

EQ. 3

$$\text{VARIANCE} = \frac{Y_1^2 + Y_2^2 + Y_3^2 + Y_4^2 + Y_5^2 + Y_6^2 - 6(\text{MEAN})^2}{5}$$

The values Y_1, Y_2, Y_3, ..., represent the results of the six analyses of each standard. The means of the low, target, and high standards are designated M_1, M_2, and M_3, respectively. The variances of the low, target, and high standards are designated V_1, V_2, and V_3, respectively. Additionally, a pooled variance, V_p, is calculated.

EQ. 4

$$V_p = \frac{\frac{V_1}{0.81} + V_2 + \frac{V_3}{1.21}}{3}$$

If the square root of V_p is less than one percent of M_2, then $M_2^2/10{,}000$ is to be used as the value of V_p in all subsequent calculations.

8.3.2.4 The test statistic shall be calculated:

EQ. 5

$$\text{TEST STATISTIC} = \frac{\left| \frac{M_3}{1.1} - \frac{M_1}{0.9} \right|}{\left(\frac{V_p}{3} \right)^{0.5}}$$

If the test statistic exceeds 2.13, then the supplier has failed to demonstrate a 20 percent difference between the high and low standards. In such a case, the standards are not acceptable.

8.3.2.5 The test statistic shall be calculated:

EQ. 6

$$\text{TEST STATISTIC} = \frac{\left| M_1 - \left(\frac{M_1}{1.6}\right) - \left(\frac{M_2}{2.2}\right) \right|}{\left(\frac{V_P}{4}\right)^{0.5}}$$

If the test statistic exceeds 2.13, the supplier has failed to demonstrate that the target standard concentration is midway between the high and low standards. In such a case, the standards are not acceptable.

8.3.2.6 The 95 percent confidence intervals for the mean result of each standard shall be calculated:

EQ. 7

$$\text{Interval for Low Standard} = M_1 \pm 2.13 \left(\frac{V_P}{6}\right)^{0.5}$$

EQ. 8

$$\text{Interval for Target Standard} = M_1 \pm 2.13 \left(\frac{V_P}{6}\right)^{0.5}$$

EQ. 9

$$\text{Interval for High Standard} = M_2 \pm 2.13 \left(\frac{V_P}{6}\right)^{0.5}$$

8.3.2.6.1 These intervals shall not overlap. If overlap is observed, then the supplier has failed to demonstrate the ability to discriminate the 10 percent difference in concentrations. In such a case, the standards are not acceptable.

8.3.2.6.2 In any event, the Contractor is responsible for the quality of the standards employed for analyses under this contract.

8.4 Requesting Standards from the USEPA Standards Repository

Solutions of analytical reference materials can be ordered from the USEPA Chemical Standards Repository, depending on availability. The Contractor may place an order for standards only after demonstrating that these standards are not available from commercial vendors, either in solution or as a neat material.

8.5 Documentation of the Verification and Preparation of Chemical Standards

It is the responsibility of the Contractor to maintain the necessary documentation to show that the chemical standards it has used in the performance of Contract Laboratory Program (CLP) analysis conform to the requirements previously listed.

8.5.1 Weighing logbooks, calculations, raw data, etc., whether produced by the Contractor or purchased from chemical supply houses, shall be maintained by the Contractor and may be subject to review during on-site inspection visits. In those cases where the documentation is supportive of the analytical results of data packages sent to USEPA, such documentation is to be kept on file by the Contractor for a period of one year.

8.5.2 Upon request by the USEPA Regional CLP Project Officer (CLP PO), the Contractor shall submit their most recent previous year's documentation (12 months) for the verification and preparation of chemical standards within 14 days of the receipt of request to the designated recipients.

8.5.3 USEPA may generate a report discussing deficiencies in the Contractor's documentation for the verification and preparation of chemical standards or may discuss the deficiencies during an on-site laboratory evaluation. In a detailed letter to the USEPA Regional CLP PO and Quality Assurance Technical Support (QATS), the Contractor shall address the deficiencies and the subsequent corrective action implemented by the Contractor to correct the deficiencies within 14 days of receipt of the report or the on-site laboratory evaluation. An alternate delivery schedule may be proposed by the Contractor, but it is the sole decision of USEPA to approve or disapprove the alternate delivery schedule. If an alternate delivery schedule is proposed, the Contractor shall describe in a letter to the USEPA Regional CLP PO and the Contracting Officer why it is unable to meet the delivery schedule listed in this section. The USEPA Regional CLP PO may grant an extension of up to 14 days for the Contractor's response letter to the standards documentation report. An extension beyond 14 days must be approved by the USEPA Contracting Officer. The Contractor shall proceed and not assume that an extension will be granted until so notified by the appropriate USEPA official.

8.5.4 If new Standard Operating Procedures (SOPs) are required to be written, or if existing SOPs are required to be rewritten or amended because of deficiencies and subsequent corrective action implemented by the Contractor, the Contractor shall write/amend the SOPs per the requirements listed in Section 6.

8.6 Corrective Actions

If the Contractor fails to adhere to the requirements listed in this section, a Contractor may expect, but USEPA is not limited to the following actions: reduction in the number of samples sent under the contract, suspension of sample shipment to the Contractor, data package audit, electronic data audit, an on-site laboratory evaluation, a remedial laboratory evaluation sample, and/or contract sanctions.

9.0 DATA PACKAGE AUDITS

9.1 Overview

Data package audits are performed by USEPA for program overview and specific Regional concerns. Standardized procedures have been established to assure uniformity of the auditing process. Data packages are periodically selected from recently received Cases. They are evaluated for the technical quality of hardcopy raw data, Quality Assurance (QA), and the adherence to contractual requirements. This function provides external monitoring of program Quality Control (QC) requirements. Data package audits are used to assess the technical quality of the data and evaluate overall laboratory performance. Audits provide USEPA with an in-depth inspection and evaluation of the Case data package with regard to achieving QA/QC acceptability. A thorough review of the raw data is completed including: all instrument readouts used for the sample results, instrument printouts, and other documentation for deviations from the contractual requirements, a check for transcription and calculation errors, a review of the qualifications of the laboratory personnel involved with the Case, and a review of the latest version of all Standard Operating Procedures (SOPs) on file.

9.2 Responding to the Data Package Audit Report

9.2.1 After completion of the data package audit, USEPA may send a copy of the data package audit report to the Contractor or may discuss the data package audit report on an on-site laboratory evaluation. In a detailed letter to the USEPA Regional Contract Laboratory Program Project Officer (CLP PO) and the USEPA designated recipient, the Contractor shall discuss the corrective actions implemented to resolve the deficiencies listed in the data package audit report within 14 days of receipt of the report.

9.2.2 An alternate delivery schedule may be proposed by the Contractor, but it is the sole decision of USEPA to approve or disapprove the alternate delivery schedule. If an alternate delivery schedule is proposed, the Contractor shall describe in a letter to the USEPA Regional CLP PO and the Contracting Officer why it is unable to meet the delivery schedule listed in this section. The USEPA Regional CLP PO may grant an extension of up to 14 days for the Contractor’s response letter to the data package report. An extension beyond 14 days must be approved by the USEPA Contracting Officer. The Contractor shall proceed and not assume that an extension will be granted until so notified by the appropriate USEPA official.

9.2.3 If new SOPs are required to be written, or if existing SOPs are required to be rewritten or amended because of deficiencies and subsequent corrective action implemented by the Contractor, the Contractor shall write/amend the SOPs per the requirements listed in Section 6.

9.3 Corrective Actions

If the Contractor fails to adhere to the requirements listed in this section, the Contractor may expect, but USEPA is not limited to the following actions: reduction in the number of samples sent under the contract, suspension of sample shipment to the Contractor, an on-site laboratory evaluation, data package audit, electronic data audit, remedial performance evaluation sample, and/or contract sanctions.

10.0 REGIONAL DATA REVIEW

10.1 Overview

Contractor data are generated to meet the specific needs of USEPA Regions. In order to verify the usability of data for the intended purpose, each Region reviews data from the perspective of the end user, based on functional guidelines for data review which have been developed jointly by the Regions and the National Program Office. Each Region uses these guidelines as the basis for data evaluation. Individual Regions may augment the basic guideline review process with additional review based on Region-specific or site-specific concerns. Regional reviews, like the sites under investigation, vary based on the nature of the problem under investigation and the Regional response appropriate to the specific circumstances.

10.1.1 Regional data reviews, relating usability of the data to a specific site, are part of the collective assessment process. They complement the review performed at the Sample Management Office (SMO), which is designed to identify contractual discrepancies, and the review performed by the National Program Office, which is designed to evaluate Contractor and method performance. These individual evaluations are integrated into a collective review that is necessary for Program and Contractor administration and management, and may be used to take appropriate action to correct deficiencies in the Contractor's performance.

11.0 PROFICIENCY TESTING

As a means of measuring and evaluating both the Contractor's and the method's analytical performance, the Contractor must participate in USEPA's Proficiency Testing Program. USEPA's Proficiency Testing Program involves the analysis of Case specific Performance Evaluation (PE) samples and the participation in interlaboratory Quarterly Blind (QB) Audits. The Contractor's analytical PE samples and QB results will be used by USEPA to assess and verify the Contractor's continuing ability to produce acceptable analytical data in accordance with the contractual requirements.

11.1 Performance Evaluation (PE) Samples

11.1.1 The PE sample(s) may be scheduled with the Contractor as frequently as on a Sample Delivery Group (SDG)-by-SDG basis. The PE samples may be sent either by the Regional client or the National Program Office. PE samples will assist USEPA in monitoring Contractor performance.

11.1.2 PE samples will be provided as either single-blinds (recognizable as a PE sample but of unknown composition), or as double-blinds (not recognizable as a PE sample and of unknown composition). The Contractor will not be informed of either the analytes/parameters or the concentrations in the PE samples.

11.1.3 The Contractor may receive the PE samples as either full volume samples or ampulated/bottled concentrates from USEPA or a designated USEPA Contractor. The PE samples shall come with instructions concerning the unique preparation procedures, if any, required to reconstitute the PE samples (i.e., the required dilution of the PE sample concentrate). PE samples are to be prepared and analyzed with the rest of the routine samples in the SDG. The Contractor shall prepare and analyze the PE sample using the procedure described in the sample preparation and method analysis sections of Exhibit D. All contract required Quality Control (QC) shall also be met. The PE

sample results are to be submitted in the SDG deliverable package per normal reporting procedures detailed in Exhibit B.

11.1.4 In addition to PE sample preparation and analysis, the Contractor shall be responsible for correctly identifying and quantitating the analytes/parameters included in each PE sample. When PE sample results are received by USEPA, the PE sample results will be evaluated for correct analytical identification and quantitation. The PE sample evaluation will be provided to the Contractor via coded evaluation sheets, by analyte/parameter. USEPA will notify the Contractor of unacceptable performance. USEPA reserves the right to adjust the PE sample acceptance windows in order to compensate for any unanticipated difficulties with a particular PE sample.

11.1.5 The Contractor shall demonstrate acceptable analytical performance for both identification and quantitation of PE sample analytes/ parameters. For unacceptable PE sample performance, USEPA may take, but is not limited to, the following actions: reduce value or rejection of data for the samples, SDG, or Case impacted, contract sanctions, reduction in the number of samples shipped to the laboratory, suspension of sample shipment, an on-site laboratory inspection, a full data package audit, electronic data audit, and/or require the laboratory to analyze a Remedial QB.

NOTE: A Contractor's prompt response demonstrating that corrective actions have been taken to ensure the Contractor's capability to meet contract requirements may facilitate continuation of full sample delivery.

11.2 Quarterly Blind (QB) Audits

11.2.1 QB Audits may be scheduled concurrently with all contract laboratories. A QB Audit is a unique analytical Case containing only PE samples (i.e., referred to as QB samples). The QB samples will be scheduled by the National Program Office through Sample Management Office (SMO). QB samples will assist USEPA in monitoring Contractor performance.

11.2.2 QB samples will be provided as single-blinds (recognizable as a PE sample but of unknown composition). The Contractor will not be informed of either the analytes or the concentrations in the PE samples.

11.2.3 The Contractor may receive the QB samples as either full volume samples or ampulated/bottled concentrates from USEPA or a designated USEPA Contractor. The QB samples shall come with instructions concerning the unique preparation procedures, if any, required to reconstitute the QB samples (i.e., the required dilution of the QB sample concentrate). The Contractor shall prepare and analyze the QB samples using the procedure described in the sample preparation and method analysis sections of Exhibit D. All contract required QC shall also be met. The QB sample results are to be submitted in the SDG deliverable package per normal reporting procedures detailed in Exhibit B.

11.2.4 In addition to QB sample preparation and analysis, the Contractor shall be responsible for correctly identifying and quantitating the analytes/parameters included in each QB sample. When QB sample results are received by USEPA, the QB sample results will be scored for correct analytical identification and quantitation. The QB sample scoring will be provided to the Contractor via coded evaluation sheets, by analyte/parameter. USEPA will notify the

Contractor of unacceptable performance. USEPA reserves the right to adjust the PE sample acceptance windows in order to compensate for any unanticipated difficulties with a particular PE sample. The Contractor's QB sample performance will be assessed into one of the following three categories:

11.2.4.1 Acceptable, No Response Required: Score greater than or equal to 90 percent. The data meets most or all of the scoring criteria. No response is required.

11.2.4.2 Acceptable, Response Explaining Deficiencies Required: Score greater than or equal to 75 percent, but less than 90 percent. Deficiencies exist in the Contractor's performance. Corrective action response required.

11.2.4.3 Unacceptable Performance, Response Explaining Deficiencies Required: Score less than 75 percent. Deficiencies exist in the Contractor's performance to the extent that the National Program Office has determined that the Contractor has not demonstrated the capability to meet the contract requirements. Corrective action response required.

11.2.5 In the case of Section 11.2.4.2 or 11.2.4.3, the Contractor shall describe the deficiency(ies) and the action(s) taken in a corrective action letter to the USEPA Regional Contract Laboratory Program Project Officer (CLP PO) and CLP Quality Assurance (QA) Coordinator within 14 days of receipt of notification from USEPA.

11.2.5.1 An alternate delivery schedule for the corrective action letter may be proposed by the Contractor, but it is the sole decision of USEPA to approve or disapprove the alternate delivery schedule. If an alternate delivery schedule is proposed, the Contractor shall describe in a letter to the USEPA Regional CLP PO and Contracting Officer why the laboratory is unable to meet the original delivery schedule listed in Section 11.2.5. The USEPA Regional CLP PO may grant an extension of up to 14 days for the Contractor's corrective action letter. An extension beyond 14 days must be approved by the USEPA Contracting Officer. The Contractor shall proceed and not assume that an extension will be granted until so notified by the appropriate USEPA official.

11.2.6 In the case of Section 11.2.4.2 or 11.2.4.3, if new Standard Operating Procedures (SOPs) are required to be written, or if existing SOPs are required to be rewritten or amended because of deficiencies and subsequent corrective action implemented by the Contractor, the Contractor shall write/amend the SOPs per the requirements listed in Section 6.

11.2.7 The Contractor shall be notified by the USEPA Contracting Officer concerning agreement or disagreement with the proposed remedy for unacceptable performance. For unacceptable QB sample performance (Section 11.2.4.3), USEPA may take, but is not limited to, the following actions: reduction in the number of samples shipped to the laboratory, suspension of sample shipment, an on-site laboratory inspection, electronic data audit, a full data package audit, and/or require the laboratory to analyze a Remedial QB sample, and/or contract sanctions.

NOTE: A Contractor's prompt response demonstrating that corrective actions have been taken to ensure the Contractor's capability to meet contract requirements may facilitate continuation of full sample delivery.

11.2.8 A Remedial QB Audit is a unique analytical Case containing only QB samples. A Remedial QB Audit may be scheduled by the National Program Office with the Contractor(s) for any of the following reasons: unacceptable PE sample performance, unacceptable QB sample performance, and/or major change in the laboratory (e.g., relocation, new owner, or high turnover of key personnel). Sections 11.2.2 through 11.2.7 apply to the Remedial QB Audit process.

11.3 Corrective Actions

If the Contractor fails to adhere to the requirements listed in this section, the Contractor may expect, but USEPA is not limited to, the following actions: reduction in the number of samples sent under the contract, suspension of sample shipment to the Contractor, a full data package audit, electronic data audit, an on-site laboratory inspection, a Remedial QB sample, and/or contract sanctions.

12.0 ON-SITE LABORATORY EVALUATIONS

12.1 Overview

As dictated by a contract laboratory's performance, the USEPA Regional Contract Laboratory Program Project Officer (CLP PO) or their authorized representative will conduct an on-site laboratory evaluation. On-site laboratory evaluations are carried out to monitor the Contractor's ability to meet selected terms and conditions specified in the contract. The evaluation process incorporates two separate categories: Quality Assurance (QA) Evaluation and an Evidentiary Audit.

12.2 Quality Assurance On-Site Evaluation

QA evaluators inspect the Contractor's facilities to verify the adequacy and maintenance of instrumentation, the continuity, experience and education of personnel, and the acceptable performance of analytical and Quality Control (QC) procedures.

12.2.1 The Contractor should expect that items to be monitored will include, but not be limited to, the following:

- Size and appearance of the facility;
- Quantity, age, availability, scheduled maintenance, and performance of instrumentation;
- Availability, appropriateness, and utilization of the Quality Assurance Plan (QAP) and Standard Operating Procedures (SOPs);
- Staff qualifications, experience, and personnel training programs;
- Reagents, standards, and sample storage facilities;
- Standard preparation logbooks and raw data;
- Bench sheets and analytical logbook maintenance and review; and
- Review of the Contractor's sample analysis/data package inspection/data management procedures.

12.2.2 Prior to an on-site evaluation, various documentation pertaining to performance of the specific Contractor is integrated in a profile package for discussion during the evaluation. Items that may be included are previous on-site reports, performance evaluation sample scores, Regional review of data, Regional QA materials, data audit reports, results of Contract Compliance Screening (CCS), and data trend reports.

12.3 Evidentiary Audit

Evidence auditors conduct an on-site laboratory evaluation to determine if laboratory policies and procedures are in place to satisfy evidence handling requirements as stated in Exhibit F. The evidence audit comprises a procedural audit, an audit of written Standard Operating Procedures (SOPs), and an audit of analytical project file documentation.

12.3.1 Procedural Audit. The procedural audit consists of review and examination of actual SOPs and accompanying documentation for the following laboratory operations: sample receiving, sample storage,

sample identification, sample security, sample tracking (from receipt to completion of analysis), analytical project file organization and assembly, and proper disposal of samples and cogenerated wastes.

12.3.2 Written SOPs Audit. The written SOPs audit consists of review and examination of the written SOPs to determine if they are accurate and complete for the following laboratory operations: sample receiving, sample storage, sample identification, sample security, sample tracking (from receipt to completion of analysis), and analytical project file organization and assembly.

12.3.3 Analytical Project File Evidence Audit. The analytical project file evidence audit consists of review and examination of the analytical project file documentation. The auditors review the files to determine:

- The accuracy of the document inventory;
- The completeness of the file;
- The adequacy and accuracy of the document numbering system;
- Traceability of sample activity;
- Identification of activity recorded on the documents; and
- Error correction methods.

12.4 Discussion of the On-Site Team's Findings

The QA and evidentiary auditors discuss their findings with the USEPA Regional CLP PO prior to debriefing the Contractor. During the debriefing, the auditors present their findings and recommendations for corrective actions necessary to the Contractor personnel.

12.5 Corrective Action Reports for Follow-Through to Quality Assurance and Evidentiary Audit Reports

Following an on-site laboratory evaluation, QA and/or evidentiary audit reports which discuss deficiencies found during the on-site evaluation may be sent to the Contractor. In a detailed letter, the Contractor shall discuss the corrective actions implemented to resolve the deficiencies discussed during the on-site evaluation and discussed in the report(s) to the USEPA Regional CLP PO within 14 days of receipt of the report.

12.5.1 An alternate delivery schedule may be proposed by the Contractor, but it is the sole decision of USEPA to approve or disapprove the alternate delivery schedule. If an alternate delivery schedule is proposed, the Contractor shall describe in a letter to the USEPA Regional CLP PO and the Contracting Officer why it is unable to meet the delivery schedule listed in this section. The USEPA Regional CLP PO may grant an extension of up to 14 days for the Contractor's response letter to the QA and evidentiary audit report. An extension beyond 14 days must be approved by the USEPA Contracting Officer. The Contractor shall proceed and not assume that an extension will be granted until so notified by the appropriate USEPA official.

12.5.2 If new SOPs are required to be written, or if existing SOPs are required to be rewritten or amended because of the deficiencies and the subsequent corrective action implemented by the Contractor, the

Contractor shall write/amend the SOPs per the requirements listed in Section 6.

12.6 Corrective Actions

If the Contractor fails to adhere to the requirements listed in this section, the Contractor may expect, but USEPA is not limited to, the following actions: reduction in the number of samples sent under the contract, suspension of sample shipment to the Contractor, an on-site laboratory evaluation, data package audit, electronic data audit, a remedial performance evaluation sample, and/or contract sanctions.

13.0 ELECTRONIC DATA AUDITS

13.1 Overview

Periodically, USEPA requests the instrument electronic data from Contractors for a specific Case in order to accomplish electronic data audits. Generally, electronic data submissions and audits are requested for the following reasons.

- Program overview;
- Indication of data quality problems;
- Support for on-site audits; and
- Specific Regional requests.

13.1.1 Depending upon the reason for an audit, the instrument electronic data from a recent Case, a specific Case, or a laboratory evaluation sample may be requested. Electronic data audits provide a mechanism to assess adherence to contractual requirements and to ensure the consistency of data reported on the hardcopy/electronic deliverables with that generated on analytical instruments. This function provides external monitoring of Program Quality Control (QC) requirements and checks adherence of the Contractor to internal Quality Assurance (QA) procedures. In addition, electronic data audits enable USEPA to evaluate the utility, precision, and accuracy of the analytical methods.

13.1.2 The Contractor shall store <u>all</u> raw and processed electronic analytical data in the appropriate instrument manufacturer's format, uncompressed, and with no security codes. The data shall include all necessary data files for a complete reconstruction of the previously submitted hardcopy and electronic deliverable data package. All associated raw data files in the instrument manufacturer proprietary software format must be submitted if those files contain data or instrumental parameters regarding any analysis and or correction applied to an instrument or analytical result. This instrument electronic data shall include data for all samples and all QC samples, including but not limited to: blanks, Matrix Spike/Matrix Spike Duplicate(s) (MS/MSD), Laboratory Control Sample (LCS), instrument performance checks [4-Bromofluorobenzene (BFB) and decafluorotriphenylphosphine (DFTPP)], initial calibrations, Continuing Calibration, as well as all Contractor-generated spectral libraries and quantitation reports required to generate the data package. In addition, the Contractor shall supply raw data for the Method Detection Limit (MDL) studies and values for the year in which the Sample Delivery Group (SDG) was analyzed. The Contractor shall maintain a written reference logbook of data files of EPA sample number, calibration data, standards, blanks, spikes, and duplicates. The logbook shall include EPA sample numbers, identified by Case and SDG.

13.1.3 The Contractor is required to retain the instrument electronic data for three years after submission of the reconciled Complete SDG File. Electronic media shipped to USEPA designated recipient must be fully usable by the recipient. Diskettes must be 3.5 inch, high density, 1.44 MB MS DOS formatted and tapes must be either 4 mm or 8 mm. Alternative means for delivery of electronic data may be utilized by the Contractor upon prior written approval by USEPA. When submitting electronic instrument data to a USEPA, the following materials shall be delivered in response to the request.

13.1.3.1 All associated raw data files for all analytical samples and all QC samples. For example, files for LCS, blanks, initial and continuing calibration standards and instrument performance check solutions (BFB and DFTPP).

13.1.3.2 All processed data files and quantitation output files associated with the raw data files described in Section 13.1.3.1.

13.1.3.3 All associated identification and calculation files used to generate the data submitted in the data package. This includes, but is not limited to, result files, acquisition files, calibration files, and method files.

13.1.3.4 All Contractor-generated Mass Spectral library files (NIST/EPA/NIH and/or Wiley, or equivalent, library not required).

13.1.3.5 A copy of the Contractor's written reference logbook relating data files to EPA sample number, LCS, BFB and DFTPP, calibration data, standards, blanks, and spikes. The logbook shall include EPA sample numbers and laboratory file identifiers for all samples, blanks, and standards, identified by Case and SDG.

13.1.3.6 A printout of the directory of all files in each directory, including all subdirectories and the files contained therein.

13.1.3.7 A copy (hardcopy) of the completed Sample Data Package.

13.1.3.8 A statement attesting to the completeness of the electronic instrument data submission, signed and dated by the Contractor's laboratory manager. The Contractor shall also provide a statement attesting that the data reported have not been altered in any way. These statements shall be part of a Cover Sheet that includes the following information relevant to the data submission:

- Contractor name;
- Date of submission;
- Case number;
- SDG number;
- Instrument make and model number for each instrument;
- Instrument operating software name and version number;
- Data software name and version used for acquisition, requantitation, and hardcopy/report generation;
- Data system computer;
- System operating software;
- Data system network;
- Data backup software;
- Data backup hardware;

- Media type and volume of data (in MB) backed up; and
- Names and telephone numbers of two Contractor contacts for further information regarding the submission.

13.2 Submission of the Instrument Electronic Data

Upon request of the USEPA Regional Contract Laboratory Program (CLP) Project Officer (CLP PO), the Contractor shall send the required instrument electronic data and all necessary documentation to USEPA designated recipient [e.g., Quality Assurance Technical Support (QATS)] within 7 days of notification. An alternate delivery schedule may be proposed by the Contractor, but it is the sole decision of USEPA to approve or disapprove the alternate delivery schedule. If an alternate delivery schedule is proposed, the Contractor shall describe in a letter to the USEPA Regional CLP PO and the Contracting Officer why it is unable to meet the delivery schedule listed in this section. The USEPA Regional CLP PO may grant an extension of up to 7 days for submission of the instrument electronic data. An extension beyond 7 days must be approved by the USEPA Contracting Officer (CO). The Contractor shall proceed and not assume that an extension will be granted until so notified by the appropriate USEPA official.

NOTE: The instrument electronic data shall be shipped according to the procedures in Exhibit F.

13.3 Responding to the Electronic Data Audit Report

After completion of the electronic data audit, USEPA may send a copy of the electronic data audit report to the Contractor or may discuss the electronic data audit report at an on-site laboratory evaluation. In a detailed letter to the USEPA Regional CLP PO, the Contractor shall discuss the corrective actions implemented to resolve the deficiencies listed in the electronic data audit report within 14 days of receipt of the report.

13.3.1 An alternate delivery schedule may be proposed by the Contractor, but it is the sole decision of USEPA to approve or disapprove the alternate delivery schedule. If an alternate delivery schedule is proposed, the Contractor shall describe in a letter to the USEPA Regional CLP PO and the Contracting Officer why it is unable to meet the delivery schedule listed in Section 13.3. The USEPA Regional CLP PO may grant an extension of up to 14 days for the Contractor's response letter to the electronic data report. An extension beyond 14 days must be approved by the USEPA Contracting Officer. The Contractor shall proceed and not assume that an extension will be granted until so notified by the appropriate USEPA official.

13.3.2 If new Standard Operating Procedures (SOPs) are required to be written or SOPs are required to be amended because of the deficiencies and the subsequent corrective action implemented by the Contractor, the Contractor shall write/amend and submit the SOPs per the requirements listed in Section 6.

13.4 Corrective Actions

If the Contractor fails to adhere to the requirements listed in Section 13, the Contractor may expect, but USEPA is not limited to, the following actions: reduction in the number of samples sent under the contract, suspension of sample shipment to the Contractor, an on-site laboratory evaluation, an electronic data audit, a data package audit, a remedial laboratory evaluation sample, and/or contract sanctions.

13.5 Maintenance of the Magnetic Tape Storage Device

13.5.1 The Contractor shall certify that the tape head alignment on the magnetic tape storage device is in compliance with the ANSI standards for nine track magnetic tapes. If the Contractor does not have documentation of alignment within the last 12 months, the Contractor must perform or have performed the manufacturer's documented head alignment procedure within 60 days of contract award. This is generally performed with a "skew" tape, certified to be in conformance with ANSI standards. The alignment must be performed by qualified personnel. The tape head alignment must be performed at a minimum once every 12 months, or whenever there is evidence that the tape head may be out of alignment.

13.5.2 The tape system, including recording head, must be in conformance with the manufacturer's physical and electrical standards. Alignment of the remaining components of the tape system such as the retracting arms, must be performed at intervals not to exceed 24 months. If the Contractor cannot demonstrate that the remaining components of the tape system are in alignment, then the Contractor must perform or have performed the manufacturer's recommended alignment procedure.

14.0 DATA MANAGEMENT

14.1 Overview

14.1.1 Data management procedures are defined as procedures specifying the acquisition or entry, update, correction, deletion, storage, and security of computer readable data and files. These procedures shall be in written form and contain a clear definition for all databases and files used to generate or resubmit deliverables. Key areas of concern include: system organization (including personnel and security), documentation operations, traceability, and Quality Control (QC).

14.1.2 Data manually entered from hardcopy shall be subject to QC checks and the error rates estimated. Systems should prevent entry of incorrect or out-of-range data and alert data entry personnel of errors. In addition, data entry error rates shall be estimated and recorded on a monthly basis by re-entering a statistical sample of the data entered and calculating discrepancy rates by data element.

14.2 Documenting Data Changes

The record of changes in the form of corrections and updates to data originally generated, submitted, and/or resubmitted shall be documented to allow traceability of updates. Documentation shall include the following for each change.

- Justification or rationale for the change.
- Date and initials of the person making the change(s). Data changes shall be implemented and reviewed by a person or group independent of the source generating the deliverable.
- Documentation of changes shall be retained according to the schedule of the original deliverable.
- Resubmitted diskettes or other deliverables shall be re-inspected as a part of the laboratory’s internal inspection process prior to resubmission. The entire deliverable, not just the changes, shall be inspected.
- The Laboratory Manager shall approve changes to originally submitted deliverables.
- Documentation of data changes may be requested by laboratory auditors.

14.3 Lifecycle Management Procedures

Lifecycle management procedures shall be applied to computer software systems developed by the Contractor to be used to generate and edit contract deliverables. Such systems shall be thoroughly tested and documented prior to utilization.

14.3.1 A software test and acceptance plan including test requirements, test results and acceptance criteria shall be developed, followed, and available in written form.

14.3.2 System changes shall not be made directly to production systems generating deliverables. Changes shall be made first to a development system and tested prior to implementation.

14.3.3 Each version of the production system will be given an identification number, date of installation, and date of last operation and will be archived.

14.3.4 System and operations documentation shall be developed and maintained for each system. Documentation shall include a user's manual and an operations and maintenance manual.

14.3.5 This documentation shall be available for on-site review and/or upon written request by the USEPA Regional Contract Laboratory Program (CLP) Project Officer (CLP PO).

14.4 Personnel Responsibilities

Individual(s) responsible for the following functions shall be identified.

- System operation and maintenance including documentation and training.
- Database integrity, including data entry, data updating and QC.
- Data and system security, backup and archiving.

EXHIBIT F

CHAIN-OF-CUSTODY, DOCUMENT CONTROL
AND WRITTEN STANDARD OPERATING PROCEDURES

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Table of Contents

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1.0 INTRODUCTION

A sample is physical evidence collected from a facility or the environment. Controlling evidence is an essential part of the hazardous waste investigation effort. To ensure that U.S. Environmental Protection Agency's (USEPA's) sample data and records supporting sample-related activities are admissible and have weight as evidence in future litigation, Contractors are required to maintain USEPA samples under Chain-of-Custody and to account for all samples and supporting records of sample handling, preparation, and analysis. Contractors shall maintain sample identity, sample custody, and all sample-related records according to the requirements in this exhibit.

1.1 Purpose of Evidence Requirements

The purpose of the evidence requirements include:

- Ensuring traceability of samples while in possession of the Contractor;
- Ensuring custody of samples while in possession of the Contractor;
- Ensuring the integrity of sample identity while in possession of the Contractor;
- Ensuring sample-related activities are recorded on documents or in other formats for USEPA sample receipt, storage, preparation, analysis, and disposal;
- Ensuring all laboratory records for each specified Sample Delivery Group (SDG) will be accounted for when the project is completed; and
- Ensuring that all laboratory records directly related to USEPA samples are assembled and delivered to USEPA or, prior to delivery, are available upon USEPA's request.

2.0 STANDARD OPERATING PROCEDURES

The Contractor shall implement the following Standard Operating Procedures (SOPs) for sample receiving, sample identification, sample security, sample storage, sample tracking and document control, computer-resident sample data control, and Complete Sample Delivery Group (SDG) File (CSF) organization and assembly to ensure accountability of USEPA sample Chain-of-Custody, as well as control of all USEPA sample-related records.

2.1 Sample Receiving

2.1.1 The Contractor shall designate a sample custodian responsible for receiving USEPA samples.

2.1.2 The Contractor shall designate a representative to receive USEPA samples in the event that the sample custodian is not available.

2.1.3 Upon receipt, the condition of shipping containers and sample containers shall be inspected and recorded on Form DC-1 by the sample custodian or a designated representative.

2.1.4 Upon receipt, the condition of the custody seals (intact/broken) shall be inspected and recorded on Form DC-1 by the sample custodian or a designated representative.

2.1.5 The sample custodian or a designated representative shall verify and record on Form DC-1, the agreement or disagreement of information recorded on all documents received with samples and information recorded on sample containers.

2.1.6 The sample custodian or a designated representative shall verify and record the following information on Form DC-1 as samples are received and inspected:

- Presence or absence and condition of custody seals on shipping and/or sample containers;
- Custody seal numbers when present;
- Condition of the sample bottles;
- Presence or absence of airbills or airbill stickers;
- Airbill or airbill sticker numbers;
- Presence or absence of Chain-of-Custody records;
- Sample tags listed/not listed on Chain-of-Custody records;
- Presence or absence of Traffic Reports (TRs) or Packing Lists;
- Presence or absence of cooler temperature indicator bottle;
- Cooler temperature;
- Date of receipt;
- Time of receipt;
- EPA sample numbers;

- Presence or absence of sample tags;
- Sample tag numbers;
- Assigned laboratory numbers;
- Samples delivered by hand; and
- Problems and discrepancies.

2.1.7 The sample custodian or a designated representative shall sign, date, and record the time on all accompanying forms, when applicable, at the time of sample receipt (e.g., Chain-of-Custody records, TRs or packing lists, and airbills).

NOTE: Initials are not acceptable.

2.1.8 The Contractor shall contact the Sample Management Office (SMO) to resolve problems and discrepancies including, but not limited to: absent documents; conflicting information; absent or broken custody seals; and unsatisfactory sample condition (e.g., leaking sample container).

2.1.9 The Contractor shall record resolution of problems and discrepancies by SMO.

2.2 Sample Identification

2.2.1 The Contractor shall maintain the identity of USEPA samples and prepared samples (including extracted samples, digested samples, and distilled samples) throughout the laboratory.

2.2.2 Each sample and sample preparation container shall be labeled with the EPA sample number or a unique laboratory sample identification number.

2.3 Sample Security

2.3.1 The Contractor shall demonstrate that USEPA sample custody is maintained from receiving through retention or disposal. A sample is in custody if:

- It is in your possession; or
- It is in your view after being in your possession; or
- It is locked in a secure area after being in your possession; or
- It is in a designated secure area, accessible only to authorized personnel.

2.3.2 The Contractor shall demonstrate security of designated secure areas.

2.4 Sample Storage

The Contractor shall designate storage areas for USEPA samples and prepared samples.

2.5 Sample Tracking and Document Control

2.5.1 The Contractor shall record all activities performed on USEPA samples.

2.5.2 Titles which identify the activities recorded shall be printed on each page of all laboratory documents. (Activities include, but are not limited to: sample receipt; sample storage; sample preparation, and sample analysis.) When a document is a record of analysis, the instrument type and parameter group (i.e., GC/MS-VOA) shall be included in the title.

2.5.3 When columns are used to organize information recorded on laboratory documents, the information recorded in the columns shall be identified in a column heading.

2.5.4 Reviewers' signatures shall be identified on laboratory documents when reviews are conducted.

2.5.5 The laboratory name shall be identified on pre-printed laboratory documents.

2.5.6 Each laboratory document entry shall be dated with the month/day/year (e.g., 01/01/2000) and signed (or initialed) by the individual(s) responsible for performing the recorded activity at the time the activity is recorded.

2.5.7 Notations on laboratory documents shall be recorded in ink.

2.5.8 Corrections to laboratory data reporting forms and raw data shall be made by drawing single lines through the errors and entering the correct information. Information shall not be obliterated or rendered unreadable. Corrections and additions to information shall be signed (or initialed) and dated.

2.5.9 Unused portions of laboratory documents shall be lined-out.

2.5.10 Pages in bound and unbound logbooks shall be sequentially numbered.

2.5.11 Instrument-specific run logs shall be maintained to enable the reconstruction of run sequences.

2.5.12 Logbook entries shall be in chronological order.

2.5.13 Logbook entries shall include only one SDG per page, except in the event where SDGs "share" Quality Control (QC) samples (e.g., instrument run logs and extraction logs).

2.5.14 Information inserted into laboratory documents shall be affixed permanently in-place. The individual responsible for inserting information shall sign and date across the insert and logbook page at the time information is inserted.

2.5.15 The Contractor shall document disposal or retention of USEPA samples, remaining portions of samples, and prepared samples.

2.6 Computer-Resident Sample Data Control

2.6.1 Contractor personnel responsible for original data entry shall be identified at the time of data input.

2.6.2 The Contractor shall make changes to electronic data in a manner which ensures that the original data entry is preserved, the editor is identified, and the revision date is recorded.

2.6.3 The Contractor shall routinely verify the accuracy of manually entered data, electronically entered data, and data acquired from instruments.

2.6.4 The Contractor shall routinely verify documents produced by the electronic data collection system to ensure accuracy of the information reported.

2.6.5 The Contractor shall ensure that the electronic data collection system is secure.

2.6.5.1 The electronic data collection system shall be maintained in a secure location.

2.6.5.2 Access to the electronic data collection system functions shall be limited to authorized personnel through utilization of software security techniques (e.g., log-ons or restricted passwords).

2.6.5.3 Electronic data collection systems shall be protected from the introduction of external programs or software (e.g., viruses).

2.6.6 The Contractor shall designate archive storage areas for electronic data and the software required to access the data.

2.6.7 The Contractor shall designate an individual responsible for maintaining archives of electronic data, including the software.

2.6.8 The Contractor shall maintain the archives of electronic data and necessary software in a secure location that shall be accessible only to authorized personnel.

2.7 Complete SDG File (CSF) Organization and Assembly

2.7.1 The Contractor shall designate a document control officer responsible for the organization and assembly of the CSF.

2.7.2 The Contractor shall designate a representative responsible for the organization and assembly of the CSF in the event that the document control officer is not available.

2.7.3 The Contractor shall maintain documents relating to the CSF in a secure location.

2.7.4 All original laboratory forms and copies of SDG-related logbook pages shall be included in the CSF.

2.7.5 Copies of laboratory documents in the CSF shall be photocopied in a manner to provide complete and legible replicates.

2.7.6 Documents relevant to each SDG including, but not limited to, the following shall be included in the CSF:

- logbook pages;
- bench sheets;
- mass spectra;
- chromatograms;
- screening records;
- preparation records;
- repreparation records;
- analytical records;
- re-analysis records;
- records of failed or attempted analysis;
- custody records;
- sample tracking records;
- raw data summaries;
- computer printouts;
- correspondence;
- FAX originals;
- library search results; and
- other.

2.7.7 The document control officer or a designated representative shall ensure that sample tags are encased in clear plastic bags before placing them in the CSF.

2.7.8 CSF documents shall be organized and assembled on an SDG-specific basis.

2.7.9 Original documents which include information relating to more than one SDG (e.g., Chain-of-Custody records, TRs, calibration logs) shall be filed in the CSF of the lowest SDG number, and copies of these originals shall be placed in the other CSF(s). The document control officer or a designated representative shall record the following statement on the copies in (indelible) dark ink:

COPY
ORIGINAL DOCUMENTS ARE INCLUDED IN CSF _____

_____
Signature

_____
Date

2.7.10 All CSFs shall be submitted with a completed Form DC-2. All resubmitted CSFs shall be submitted with a new or revised Form DC-2.

2.7.11 Each item in the CSF and resubmitted CSFs shall be inventoried and assembled in the order specified on Form DC-2. Each page of the CSF shall be stamped with a sequential number. Page number ranges shall be recorded in the columns provided on Form DC-2. Intentional gaps in the page numbering sequence shall be recorded in the "Comments" section on Form DC-2. When inserting new or inadvertently omitted documents, the Contractor shall identify them with unique accountable numbers. The unique accountable numbers and the locations of the documents shall be recorded in the "Other Records" section on Form DC-2.

2.7.12 Before shipping each CSF, the document control officer or a designated representative shall verify the agreement of information recorded on all documentation and ensure that the information is consistent and the CSF is complete.

2.7.13 The document control officer or a designated representative shall document the shipment of deliverable packages including what was sent, to whom the packages were sent, the date, and the carrier used.

2.7.14 Shipments of deliverable packages, including re-submittals, shall be sealed with custody seals by the document control officer or a designated representative in a manner such that opening the packages would break the seals.

2.7.15 Custody seals shall be signed and dated by the document control officer or a designated representative when sealing deliverable packages.

3.0 WRITTEN STANDARD OPERATING PROCEDURES

The Contractor shall develop and implement the following written Standard Operating Procedures (SOPs) for sample receiving, sample identification, sample security, sample storage, sample tracking and document control, computer-resident sample data control, and Complete Sample Delivery Group (SDG) File (CSF) organization and assembly to ensure accountability for USEPA sample Chain-of-Custody and control of all USEPA sample-related records.

3.1 Sample Receiving

3.1.1 The Contractor shall have written SOPs for sample receiving which accurately reflect the procedures used by the laboratory.

3.1.2 The written SOPs for sample receiving shall ensure that the procedures listed below are in-use at the laboratory.

3.1.2.1 The condition of shipping containers and sample containers are inspected and recorded on Form DC-1 upon receipt by the sample custodian or a designated representative.

3.1.2.2 The condition of custody seals are inspected and recorded on Form DC-1 upon receipt by the sample custodian or a designated representative.

3.1.2.3 The presence or absence of the following documents/items accompanying the sample shipment is verified and recorded on Form DC-1 by the sample custodian or a designated representative:

- Custody seals;
- Chain-of-Custody records;
- Traffic Reports (TRs) or Packing Lists;
- Airbills or airbill stickers;
- Sample tags; and
- Cooler temperature indicator bottle.

3.1.2.4 The agreement or disagreement of information recorded on shipping documents with information recorded on sample containers is verified and recorded on Form DC-1 by the sample custodian or a designated representative.

3.1.2.5 The following information is recorded on Form DC-1 by the sample custodian or a designated representative as samples are received and inspected:

- Presence or absence and condition of custody seals on shipping and/or sample containers;
- Custody seal numbers when present;
- Condition of the sample bottles;
- Presence or absence of airbills or airbill stickers;
- Airbill or airbill sticker numbers;

- Presence or absence of Chain-of-Custody records;
- Sample tags listed/not listed on Chain-of-Custody records;
- Presence or absence of TRs or Packing Lists;
- Presence or absence of cooler temperature indicator bottle;
- Cooler temperature;
- Date of receipt;
- Time of receipt;
- EPA sample numbers;
- Presence or absence of sample tags;
- Sample tag numbers;
- Assigned laboratory numbers;
- Samples delivered by hand; and
- Problems and discrepancies.

3.1.2.6 The sample custodian or a designated representative shall sign, date, and record the time on all accompanying forms, when applicable, at the time of sample receipt (e.g., Chain-of-Custody records, TRs or packing lists, and airbills).

NOTE: Initials are not acceptable.

3.1.2.7 The Contractor shall contact the Sample Management Office (SMO) to resolve problems and discrepancies including, but not limited to: absent documents; conflicting information; absent or broken custody seals; and unsatisfactory sample condition (e.g., leaking sample container).

3.1.2.8 The Contractor shall record resolution of problems and discrepancies by SMO.

3.2 Sample Identification

3.2.1 The Contractor shall have written SOPs for sample identification which accurately reflect the procedures used by the laboratory.

3.2.2 The written SOPs for sample identification shall ensure that the procedures listed below are in use at the laboratory.

3.2.2.1 The identity of USEPA samples and prepared samples is maintained throughout the laboratory when:

- The Contractor assigns unique laboratory sample identification numbers, thus the written SOPs shall include a description of the procedure used to assign these numbers;
- The Contractor uses prefixes or suffixes in addition to laboratory sample identification numbers, thus the written SOPs shall include their definitions; and

- The Contractor uses methods to uniquely identify fractions/parameter groups and matrix type, thus the written SOPs shall include a description of these methods.

3.2.2.2 Each sample and sample preparation container is labeled with the EPA sample number or a unique laboratory sample identification number.

3.3 Sample Security

3.3.1 The Contractor shall have written SOPs for sample security which accurately reflect the procedures used by the laboratory.

3.3.2 The written SOPs for sample security shall include the items listed below.

3.3.2.1 Procedures which ensure the following:

- Sample custody is maintained; and
- The security of designated secure areas is maintained.

3.3.2.2 A list of authorized personnel who have access to locked storage areas.

3.4 Sample Storage

3.4.1 The Contractor shall have written SOPs for sample storage which accurately reflect the procedures used by the laboratory.

3.4.2 The written SOPs for sample storage shall describe locations, contents, and identities of all storage areas for USEPA samples and prepared samples in the laboratory.

3.5 Sample Tracking and Document Control

3.5.1 The Contractor shall have written SOPs for sample tracking and document control which accurately reflect the procedures used by the laboratory.

3.5.2 The written SOPs for sample tracking and document control shall include the items listed below.

3.5.2.1 Examples of all laboratory documents used during sample receiving, sample storage, sample transfer, sample analyses, CSF organization and assembly, and sample retention or disposal.

3.5.2.2 Procedures which ensure the following:

- All activities performed on USEPA samples are recorded;
- Titles which identify the activities recorded are printed on each page of all laboratory documents;
- Information recorded in columns is identified with column headings;
- Reviewers' signatures are identified on laboratory documents;
- The laboratory name is included on pre-printed laboratory documents;

- Laboratory document entries are signed and dated with the month/day/year (e.g., 01/01/2000);
- Entries on all laboratory documents are recorded in ink;
- Corrections and additions to laboratory documents are made by drawing single lines through the errors, entering the correct information, and initialing and dating the new information;
- Unused portions of laboratory documents are lined-out;
- Pages in bound and unbound logbooks are sequentially numbered;
- Instrument-specific run logs are maintained to enable the reconstruction of run sequences;
- Logbook entries are recorded in chronological order;
- Entries are recorded for only one SDG per page, except in the event where SDGs "share" Quality Control (QC) samples (e.g., instrument run logs and extraction logs);
- Information inserted in laboratory documents is affixed permanently, signed or initialed, and dated across the insert; and
- The retention or disposal of USEPA samples, remaining portions of samples, and prepared samples is documented.

3.6 Computer-Resident Sample Data Control

3.6.1 The Contractor shall have written SOPs for computer-resident sample data control which accurately reflect the procedures used by the laboratory.

3.6.2 The written SOPs for computer-resident sample data control shall include the items listed below.

3.6.2.1 Procedures which ensure the following:

- Contractor personnel responsible for original data entry are identified;
- Changes to electronic data are made such that the original data entry is preserved, the editor is identified, and the revision date is recorded;
- The accuracy of manually entered data, electronically entered data, and data acquired from instruments is verified;
- Report documents produced by the electronic data collection system are routinely verified to ensure the accuracy of the information reported;
- Electronic data collection system security is maintained;
- Archives of electronic data and accompanying software are maintained in a secure location; and
- Off-site backup and storage of electronic data is maintained.

3.6.2.2 Descriptions of archive storage areas for the electronic data and the software required to access data archives.

3.6.2.3 A list of authorized personnel who have access to electronic data collection system functions and to archived data.

3.7 CSF Organization and Assembly

3.7.1 The Contractor shall have written SOPs for CSF organization and assembly which accurately reflect the procedures used by the laboratory.

3.7.2 The written SOPs for CSF organization and assembly shall ensure that the procedures listed below are in-use at the laboratory.

- Documents relating to the CSF are maintained in a secure location;
- All original laboratory forms and copies of SDG-related logbook pages are included in the CSF;
- Laboratory documents are photocopied in a manner to provide complete and legible replicates;
- All documents relevant to each SDG are included in the CSF;
- Sample tags are encased in clear plastic bags by the document control officer or a designated representative before being placed in the CSF;
- The CSF is organized and assembled on an SDG-specific basis;
- Original documents which contain information relating to more than one SDG are filed in the CSF of the lowest SDG and copies are referenced to originals in the event that an original document contains information relating to more than one SDG;
- Each CSF is submitted with a completed Form DC-2, and re-submitted CSFs are submitted with a new or revised Form DC-2;
- Each page of the CSF is stamped with a sequential number and the page number ranges are recorded in the columns provided on Form DC-2. Intentional gaps in the page numbering sequence are recorded in the "Comments" section of Form DC-2. Inserted documents are recorded in the "Other Records" section of Form DC-2;
- Consistency and completeness of the CSF are verified by the document control officer or a designated representative;
- Shipments of deliverable packages are documented by the document control officer or a designated representative;
- Deliverable packages are shipped by the document control officer or a designated representative using custody seals in a manner such that opening the packages would break the seals; and
- Custody seals are signed and dated by the document control officer or a designated representative before placing them on deliverable packages.

EXHIBIT G

GLOSSARY OF TERMS

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ALIQUOT - A measured portion of a field sample, standard, or solution, taken for sample preparation and/or analysis.

ANALYSIS DATE/TIME - The date and military time (24-hour clock) of the injection of the sample, standard, or blank into the GC/MS or GC system.

BAR GRAPH SPECTRUM - A plot of the mass-to-charge ratio (m/e) versus relative intensity of the ion current.

BLANK - An analytical sample designed to assess specific sources of contamination. See individual definitions for types of blanks.

BREAKDOWN - A measure of the decomposition of certain analytes (DDT and Endrin) into by-products.

4-BROMOFLUOROBENZENE (BFB) - The compound chosen to establish mass spectral instrument performance for volatile (VOA) analyses.

CALIBRATION FACTOR (CF) - A measure of the gas chromatographic response of a target analyte to the mass injected.

CASE - A finite, usually predetermined number of samples collected over a given time period from a particular site. Case numbers are assigned by the Sample Management Office (SMO). A Case consists of one or more Sample Delivery Groups (SDGs).

CONTAMINATION - A component of a sample or an extract that is not representative of the environmental source of the sample. Contamination may stem from other samples, sampling equipment, while in transit, from laboratory reagents, laboratory environment, or analytical instruments.

CONTINUING CALIBRATION - Analytical standard run every 12 hours to verify the initial calibration of the system.

CONTINUOUS LIQUID-LIQUID EXTRACTION - Used herein synonymously with the terms continuous extraction, continuous liquid extraction, and liquid extraction. This extraction technique involves boiling the extraction solvent in a flask and condensing the solvent above the aqueous sample. The condensed solvent drips through the sample, extracting the compounds of interest from the aqueous phase.

DATE - MM/DD/YYYY - where MM = 01 for January, 02 for February, ... 12 for December; DD = 01 to 31; YYYY = 1998, 1999, 2000, 2001, etc.

DAY - Unless otherwise specified, day shall mean calendar day.

DECAFLUOROTRIPHENYLPHOSPHINE (DFTPP) - Compound chosen to establish mass spectral instrument performance for semivolatile analysis.

DEUTERATED MONITORING COMPOUNDS (DMCs) - Compounds added to every calibration standard, blank, and sample used to evaluate the efficiency of the extraction/purge and trap procedures, and the performance of the gas chromatograph/mass spectrometer (GC/MS) systems. DMCs are isotopically labeled (deuterated) analogs of native target compounds. DMCs are not expected to be naturally detected in the environmental media.

EXTRACTABLE - A compound that can be partitioned into an organic solvent from the sample matrix and is amenable to gas chromatography. Extractables include semivolatile (SVOA) and pesticide/Aroclor compounds.

EXTRACTED ION CURRENT PROFILE (EICP) - A plot of ion abundance versus time (or scan number) for ion(s) of specified mass(es).

FIELD SAMPLE - A portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

GAS CHROMATOGRAPH (GC) - The instrument used to separate analytes on a stationary phase within a chromatographic column. The analytes are volatized directly from the sample (VOA), or injected as extracts (SVA and PEST). In VOA and SVA analysis, the compounds are detected by a Mass Spectrometer. In PEST analysis, the compounds are detected by an Electron Capture Detector.

IN-HOUSE - At the Contractor's facility.

INITIAL CALIBRATION - Analysis of analytical standards for a series of different specified concentrations; used to define the linearity and dynamic range of the response of the mass spectrometer or electron capture detector to the target compounds.

INTEGRATION SCAN RANGE - The scan number of the scan at the beginning of the area of integration to the scan number at the end of the area of integration. Performed in accordance with Exhibit D VOA and SVOA.

INTEGRATION TIME RANGE - The retention time at the beginning of the area of integration to the retention time at the end of the area of integration.

INTERNAL STANDARDS - Compounds added to every standard, blank, sample (for volatiles), sample extract (for semivolatiles), including Laboratory Control Sample, at a known concentration, prior to analysis. Internal standards are used as the basis for quantitation of the target compounds.

INSTRUMENT BLANK - A blank designed to determine the level of contamination associated with the analytical instruments.

LABORATORY - Synonymous with Contractor as used herein.

LABORATORY CONTROL SAMPLE (LCS) - The LCS is an internal laboratory quality control sample designed to assess (on an SDG-by-SDG basis) the capability of the contractor to perform the analytical method.

m/z - Mass to charge ratio, synonymous with "m/e".

MATRIX - The predominant material of which the sample to be analyzed is composed. For the purpose of this SOW, the sample matrix is water.

MATRIX EFFECT - In general, the effect of a particular matrix (water) on the constituents with which it contacts. Matrix effects may prevent efficient purging/extraction of target analytes, and may affect DMC and surrogate recoveries. In addition, nontarget analytes may be extracted from the matrix causing interferences.

MATRIX SPIKE - Aliquot of the water sample fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

MATRIX SPIKE DUPLICATE - A second aliquot of the same sample as the matrix spike (above) that is spiked in order to determine the precision of the method.

METHOD BLANK - An analytical control consisting of all reagents, internal standards, and surrogate standards (or DMCs for VOA and SV), that is carried throughout the entire analytical procedure. The method blank is used to define the level of laboratory, background, and reagent contamination.

NARRATIVE (SDG Narrative) - Portion of the data package which includes laboratory, contract, Case and sample number identification, and descriptive documentation of any problems encountered in processing the samples, along with corrective action taken and problem resolution. Complete SDG Narrative specifications are included in Exhibit B.

PERCENT DIFFERENCE (%D) - As used in this SOW and elsewhere to compare two values, the percent difference indicates both the direction and the magnitude of the comparison, i.e., the percent difference may be either negative, positive, or zero.

PERFORMANCE EVALUATION MIXTURE - A calibration solution of specific analytes used to evaluate both recovery and percent breakdown as measures of performance.

PERFORMANCE EVALUATION SAMPLE - An external quality control sample prepared by USEPA and is designed to assess the capability of the Contractor to perform the analytical method.

PRIMARY QUANTITATION ION - A contract specified ion used to quantitate a target analyte.

PROTOCOL - Describes the exact procedures to be followed with respect to sample receipt and handling, analytical methods, data reporting and deliverables, and document control. Used synonymously with Statement of Work (SOW).

PURGE AND TRAP (DEVICE) - Analytical technique (device) used to isolate volatile (purgeable) organics by stripping the compounds from water by a stream of inert gas, trapping the compounds on an adsorbent such as a porous polymer trap, and thermally desorbing the trapped compounds onto the gas chromatographic column.

PURGEABLES - Volatile compounds.

RECONSTRUCTED ION CHROMATOGRAM (RIC) - A mass spectral graphical representation of the separation achieved by a gas chromatograph; a plot of total ion current versus retention time.

RELATIVE PERCENT DIFFERENCE (RPD) - As used in this SOW and elsewhere to compare two values, the relative percent difference is based on the mean of the two values.

RELATIVE RESPONSE FACTOR (RRF) - A measure of the relative mass spectral response of an analyte compared to its internal standard. RRFs are determined by analysis of standards and are used in the calculation of concentrations of analytes in samples.

RELATIVE RETENTION TIME (RRT) - The ratio of the retention time of a compound to that of a standard (such as an internal standard).

RESOLUTION - Also termed separation or percent resolution, the separation between peaks on a chromatogram, calculated by dividing the depth of the valley between the peaks by the peak height of the smaller peak being resolved, multiplied by 100.

RESOLUTION CHECK MIXTURE - A solution of specific analytes used to determine resolution of adjacent peaks; used to assess instrumental performance.

RESPONSE - or Instrumental Response - A measurement of the output of the GC detector (MS, or EC) in which the intensity of the signal is proportionate to the amount (or concentration) detected. Measured by peak area or peak height.

RETENTION TIME (RT) - The time a target analyte is retained on a GC column before elution. The identification of a target analyte is dependent on a target compound's retention time falling within the specified retention time window established for that compound. Retention time is dependent on the nature of the column's stationary phase, column diameter, temperature, flow rate, and other parameters.

SAMPLE DELIVERY GROUP (SDG) - A unit within a sample Case that is used to identify a group of samples for delivery. An SDG is defined by the following, whichever is most frequent:

- Each Case of field samples received, or
- Each 20 field samples [excluding Performance Evaluation (PE) samples] within a Case, or
- Each 7 calendar day period during which field samples in a Case are received (said period beginning with the receipt of the first sample in the SDG).

In addition, all samples and/or sample fractions assigned to an SDG must have been scheduled under the same contractual turnaround time. Preliminary Results have no impact on defining the SDG.

SAMPLE MANAGEMENT OFFICE (SMO) - A contractor operated facility operated under the Contract Laboratory Analytical Services Support (CLASS) contract, awarded and administered by USEPA.

SAMPLE NUMBER (EPA Sample Number) - A unique identification number designated by USEPA to each sample. The EPA sample number appears on the sample Traffic Report (TR) which documents information on that sample.

SECONDARY QUANTITATION ION - Contract specified ion(s) to be used in quantitation of target analytes when interferences prevent the use of the primary quantitation ion.

SEMIVOLATILE (SV) COMPOUNDS - Compounds amenable to analysis by extraction of the sample with an organic solvent. Used synonymously with Base/Neutral/Acid (BNA) compounds.

STANDARD ANALYSIS - An analytical determination made with known quantities of target compounds; used to determine response factors.

STORAGE BLANK - Reagent water (two 40.0 mL aliquots) stored with volatile samples in an SDG. It is analyzed after all samples in that SDG have been analyzed; and it is used to determine the level of contamination acquired during storage.

SULFUR CLEANUP BLANK - A modified method blank that is prepared only when some of the samples in a batch are subjected to sulfur cleanup. It is used to determine the level of contamination associated with the sulfur cleanup procedure. When all of the samples are subjected to sulfur cleanup, then the method blank serves this purpose. When none of the samples are subjected to sulfur cleanup, no sulfur cleanup blank is required.

SURROGATES (Surrogate Standard) - For pesticides/Aroclors, compounds added to every blank, sample, including Laboratory Control Sample, requested MS/MSD, and standard; used to evaluate analytical efficiency by measuring recovery. Surrogates are not expected to be detected in environmental media.

TARGET COMPOUND LIST (TCL) - A list of compounds designated by the Statement of Work (Exhibit C) for analysis.

TENTATIVELY IDENTIFIED COMPOUNDS (TIC) - Compounds detected in samples that are not target compounds, internal standards, deuterated monitoring compounds, or surrogates. Up to 30 peaks, not including those identified as alkanes (those greater than 10% of the peak area or height of the nearest internal standard), are subjected to mass spectral library searches for tentative identification.

TIME - When required to record time on any deliverable item, time shall be expressed as Military Time, i.e., a 24-hour clock (0000-2359).

TRAFFIC REPORT (TR) - A USEPA sample identification form filled out by the sampler, which accompanies the sample during shipment to the laboratory and which documents sample condition and receipt by the laboratory.

TWELVE-HOUR TIME PERIOD - The twelve (12)-hour time period for GC/MS system instrument performance check, standards calibration (initial or continuing calibration), and method blank analysis begins at the moment of injection of the DFTPP or BFB analysis that the laboratory submits as documentation of instrument performance. The time period ends after 12 hours have elapsed according to the system clock. For pesticide/Aroclor analyses performed by GC/EC, the 12-hour time period in the analytical sequence begins at the moment of injection of the instrument blank that precedes sample analyses, and ends after twelve hours have elapsed according to the system clock.

VALIDATED TIME OF SAMPLE RECEIPT (VTSR) - The date on which a sample is received at the Contractor's facility, as recorded on the shipper's delivery receipt and Sample Traffic Report.

VOLATILE COMPOUNDS - Compounds amenable to analysis by the purge and trap technique. Used synonymously with purgeable compounds.

WIDE BORE CAPILLARY COLUMN - A gas chromatographic column with an internal diameter (ID) that is greater than or equal to 0.53 mm. Columns with lesser diameters are classified as narrow bore capillary columns.

EXHIBIT H

AGENCY STANDARD IMPLEMENTATION

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1.0 FORMAT CHARACTERISTICS

1.1 This constitutes an implementation of the USEPA Agency Standard for Electronic Data Transmission based upon analytical results and ancillary information required by the contract. All data generated by a single analysis are grouped together, and the groups are aggregated to produce files that report data from a Sample Delivery Group (SDG). Because this implementation is only a subset of the Agency Standard, some fields have been replaced by delimiters as place holders for non-Contract Laboratory Program (non-CLP) data elements.

1.2 This implementation includes detailed specifications for the required format of each record. The position in the record where each field is to be contained relevant to other fields is specified, as well as the maximum length of the field. Each field's required contents are specified as literal (contained in quotes), which must appear exactly as shown (without quotes), or as a variable for which format and/or descriptions are listed in the format/contents column. Options and examples are listed for most fields. For fields where more than three options are available, a list and description of options are supplied on a separate page following the record descriptions. Fields are separated from each other by the delimiter "|" (ASCII 124). Fields that do not contain data should be zero length or a blank field (empty with no space or additional delimiters between the delimiters before and after the field) with the delimiter as a place holder. For the purposes of Section 9 of this exhibit, wherever "blank" is given as an option under the "Format/Contents" column, it refers to a blank field as explained above.

1.3 Numeric fields may contain numeric digits, a decimal place, and a leading minus sign. A positive sign is assumed if no negative sign is entered in a numeric field and shall not be entered into any numeric field. Values that exceed the maximum length allowed shall be reported to the maximum possible, maintaining the specified decimal place and maximum field length restrictions.

1.4 Requirements for significant figures and number of decimal places are specified in Exhibit B. The numeric field lengths are specified such that all possible numeric values can be written to the file. The size of the numeric field indicates the maximum number of digits, including a decimal place and negative sign (if appropriate), that can appear in the field at the same time. Therefore, the number reported may need to be rounded (using rounding rules described in Exhibit B) to fit into the field. The rounding shall maintain the greatest significance possible providing the field length limitation. In addition, the rounded number that appears on the form, and therefore in the field on the diskette file, must be used in any calculation that may result in other numbers reported on the same form or other forms in the SDG. The numbers/values reported by the Contractor are used by CCS to calculate a result [e.g., Contract Required Quantitation Limit (CRQL)]. The final value calculated by CCS is then rounded according to rounding rules described in Exhibit B and is used for comparison to the final value (e.g., CRQL) reported by the Contractor. Field lengths should only be as long as necessary to contain the data; packing with blanks is not allowed.

1.5 USEPA is currently developing a data delivery strategy that may be used as an alternative to the requirements stated in Exhibit H. This strategy’s intent is to provide a neutral data delivery structure to the Contractor that will further facilitate the exchange of analytical information generated under this analytical protocol. The proposed strategy is intended to accommodate laboratories that generate data transmission files under multiple data formats. Upon implementation of

this alternate electronic data delivery strategy by the CLP and prior to submission of data in alternate format(s), the Contractor must first demonstrate its ability to provide electronic data as stated in this Exhibit H, and obtain written permission from the CLP for the submission of data in alternate format(s). The Contractor will receive a written response to its request within 90 calendar days. However, until the implementation of this alternate electronic data delivery strategy by the CLP, all electronic data deliverables must be provided as specified in this Exhibit H.

2.0 RECORD TYPES

2.1 The Agency Standard consists of variable length ASCII records. Maximum field length specifications match the reporting requirements in Exhibit B. The last two bytes of each record shall contain "carriage return" and "line feed", respectively.

2.2 This implementation consists of twelve record types that can be summarized in four groups, designated by the first record type in each group:

Type	Type ID	Contents
Run Header	10	Information pertinent to a group of samples processed in a continuous sequence; usually several per SDG
Sample Header	20	Sample identifying, qualifying, and linking information
Results Record	30	Analyte results and qualifications
Comments Record	90	Free form comments

2.3 A separate run header is used for volatiles (VOA), semivolatiles (SV), and for each column analysis for pesticides (PEST) [minimum of four type 10 series for VOA/SV/PEST Sample Delivery Group (SDG)]. The 20 series records contain sample characteristics and link samples within an SDG to the corresponding calibrations, blanks, and other Quality Controls (QCs). The 30 series records contain the actual analytical results by analyte within each sample. The 10, 20, and 30 records are associated with each other by their position in the file (i.e., 30 series records follow the corresponding 20 series, which in turn follow the 10 series run header records).

3.0 PRODUCTION RUNS

3.1 A production run represents a "group" or "batch" of samples that are processed in a continuous sequence under relatively stable conditions. Specifically:

3.1.1 Calibration - All samples in a run use the same initial calibration data.

3.1.2 Method number - Constant throughout a run.

3.1.3 Instrument conditions - Constant throughout a run.

3.2 Each instrumental analysis consists of a separate production run and is reported in a separate file. There will be a separate production run for each of the two pesticide Gas Chromatograph (GC) columns utilized. Thus, a full three fraction analysis will consist of a minimum of four production runs.

3.3 Example of the Sequence of Record Types in a File

10			Contains Run Header information.
	11		Contains additional run-wide information.
	20		Occurs once for each sample, calibration, mean response factor, matrix spike duplicate result, etc. Acts as a header.
	21		
	22		Contains additional information for samples.
	23		
	27		
		30	Occurs once for each final analytical result. Reports the value being determined as defined by the type 20.
		32	Reports any auxiliary data necessary.
		33	Reports compound names for tentatively identified compounds (TICs) if necessary.
		36	Reports any instrumental data necessary.
		30	Values for the next analyte or parameter being measured.
		32	Additional data may vary for each parameter, and may
		33	occur in any order. Multiple occurrences of the same
		36	record type, however, must be consecutive.
		30	Continues for as many as are necessary.
		32	
		33	
		36	
		30	
		32	
		33	
		36	
	20		Next Sample Header record. The following applies to the
	21		next sample or other group of data.
	22		
		30	
		32	
		33	
		36	
		30	
		32	
		33	
		36	

etc.

20
21
30
32
33
36
etc.

4.0 RECORD SEQUENCE

4.1 The sequence of records for Agency Standard files is as follows: A Run Header (type 10) record shall be present once and once only (per file) as the first record in a file. Therefore, a complete VOA/SV/PEST SDG will consist of several files.

4.2 Each environmental sample, calibration standard, or Quality Control (QC) sample is represented by a group composed of type 20, 21, 22, 23, and 27 records, that hold sample level identifying information, followed by type 30, 32, 33, and 36 records for each method analyte including surrogates, deuterated monitoring compounds, and internal standards in the sample. The type 20 record holds a count for the number of method analytes being determined and includes all target compounds, surrogates, deuterated monitoring compounds, and internal standards plus each peak of the multi-component pesticides [do not include Tentatively Identified Compounds (TICs) in this count]. A separate field on the type 23 record contains the number of TICs found. Type 20 records shall occur in the order of sample analysis. In addition, a type 20 record with a QC code "MNC", followed by a type 30 record for each method analyte (reporting values such as mean response factors) will appear after the type 10 or type 11 record and before the type 20 record that initiates the analytical sequence. Similarly, for pesticide runs, a type 20 record with a Quality Control (QC) code "FLO" for Florisil recovery, followed by type 30 records for each of the method analytes (and the two surrogates) included in the Florisil check will appear before the type 20 record that initiates the analytical sequence.

4.3 Type 90 comment records may be defined to occupy any position after the type 10 (header) record.

5.0 FILE/RECORD INTEGRITY

All record types shall contain the following check fields to ensure file and record integrity:

Record Position	Field Length	Field Contents	Remarks
First Field	2	Record type	"10" or as appropriate
Last Field	5	Record sequence number	00001-99999, numbered within file sequentially
	4	Record checksum[1]	Four hexadecimal digits
	2	Must contain CR and LF	

6.0 DATES AND TIMES

Date or time-of-day information consists of successive groups of two decimal digits (except year, which is four decimal digits), each separated by delimiters. Dates are given in the order YYYY MM DD, and times as HH MM. All hours shall be given as 00 to 23 using a 24-hour clock and shall be local time. All days shall be given as 01 to 31. All months shall be given as 01 to 12 (e.g., 01 is January, 02 is February).

7.0 MULTIPLE VOLUME DATA

There is no requirement under this format that all the data from an entire Sample Delivery Group (SDG) fit onto a single diskette. However, each single production run must fit onto a single diskette if possible. If that is not possible, then it is necessary that all files start with a type 10 record, and that the multiple type 10 records for each file of the same production run be identical. Information for a single sample shall not be split between files.

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[1]The checksum is the sum of the ASCII representation of the data on the record up to the Record Sequence Number (not including the Record Sequence Number) plus the checksum of the previous record. The sum is taken modulo 65536 (2^{16}) and is represented as four hexadecimal digits (i.e., the remainder of the sum divided by 65536 represented as four hexadecimal digits).

8.0 DELIVERABLE

8.1 The file shall be submitted on IBM-compatible, 3.5 inch high density 1.44 MB diskettes. The diskettes shall be formatted and recorded using MS-DOS Operating System. The diskettes shall contain all information relevant to one and only one Sample Delivery Group (SDG). An alternative means of electronic transmission may be utilized if approved in advance by the USEPA.

8.2 Agency Standard data from an entire SDG may not fit onto a single diskette. If a single production run is being split onto multiple diskettes, then all files shall start with a type 10 record, and the multiple type 10 records for each file of the same production run shall be identical. Do not split the data from a single sample onto multiple diskettes.

8.3 Information on the diskette **must correspond** to information submitted in the hardcopy raw data package and on the hardcopy raw data package forms. For example, type 30 results field specifies maximum length of 13. When reporting Contract Required Quantitation Limits (CRQLs) or results on Form 1, maximum length is 13 as is specified in this exhibit; when reporting 'calculated amounts' on Form 7D, hardcopy specified maximum length is 8. Unused records shall not be included on the diskettes. If the information submitted in the hardcopy data package forms is changed, the information in the electronic file (e.g., diskette) shall be changed accordingly, and a complete electronic deliverable containing all the information for the SDG shall be resubmitted along with the hardcopy at no additional cost to the USEPA.

8.4 Each diskette shall be identified with an external label containing (in this order) the following information:

- Disk Density;
- File Name(s);
- Laboratory Name (optional);
- Laboratory Code;
- Contract Number;
- Case Number/SDG;
- Client Number (where applicable); and
- Initial Submission or Resubmission (as applicable) and Date.

8.5 The format for File Name shall be XXXXX.O01 to XXXXX.O99. Where XXXXX is the SDG identifier, O designates Organics, and 01 through 99 is the file number.

8.6 Dimensions of the label must be in the range of 2-1/2" to 2-3/4" long by 2" to 2-1/8" wide for a 3-1/2 inch IBM-compatible diskette.

8.7 Section 9.0 (Record Listing) provides information for the usage of each of the record types. Where specified, labels indicate the nature of the value(s) that follow on that record. If the value(s) will not be reported, the label shall be omitted.

8.7.1 A record type 30 for each Target Compound List (TCL) compound, surrogate, deuterated monitoring compound, and internal standard shall be reported. If the TCL is not detected, the 'U' qualifier in the appropriate field shall be indicative of that.

8.7.2 For multicomponent analytes (Aroclors/toxaphene), if the multicomponent analyte is detected, a record type 30 and 32 shall be reported for each peak identified.

9.0 RECORD LISTING

The following lists every record type required to report data from a single Sample Delivery Group (SDG).

9.1 Production Run Header Record (Type 10)

Use: Each production run will start with a record type 10.

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"10"
6	Delimiters	||||||
5	INSTRUMENT/DETECTOR	Character[2]
1	Delimiter	|
8	METHOD NUMBER	Character[3]
2	Delimiters	||
6	LAB CODE	Character
4	Delimiters	||||
11	CONTRACT NUMBER	Character
1	Delimiter	|
10	INSTRUMENT ID	Character
2	Delimiters	||
25	LABORATORY NAME	Character
2	Delimiters	||
5	RECORD SEQUENCE NUMBER	Numeric
4	CHECKSUM	Character

[2]General descriptor (GC/MS for VOA/SV analysis or GC for pesticide analysis on GC/ECD).

[3]OLC03.1V for volatiles; OLC03.1B for semivolatiles; OLC03.1P for pesticides. (O for Organic, L for Low, C for Concentration, zero three point zero for document number, V for volatiles, B for semivolatiles, P for pesticides.)

9.2 Chromatography Record (Type 11)

Use: To describe chromatograph condition. Must be present once for each production run immediately following the record type 10.

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"11"
1	Delimiter	|
10	GC COLUMN IDENTIFICATION	Character
2	Delimiters	||
4	GC COLUMN ID[4]	Numeric (mm)
11	Delimiters	|||||||||||
5	RECORD SEQUENCE NO.	Numeric
4	CHECKSUM	Character

[4]Internal Diameter of the GC column used.

9.3 Sample Header Data Record (Type 20)

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"20"
2	Delimiters	||
12	EPA SAMPLE NUMBER	As is exactly on the hardcopy form
1	Delimiter	|
1	MATRIX	CHARACTER[5]
1	Delimiter	|
3	QC CODE	Character (See Section 10)
1	Delimiter	|
3	SAMPLE QUALIFIER	RIN/REX/REJ/SRN/blank[6]
1	Delimiter	|
5	CASE NUMBER	Numeric
1	Delimiter	|
6	SDG NO.	Character
1	Delimiter	|
4	SAMPLE/BLANK/STANDARDS YEAR ANALYZED	YYYY
1	Delimiter	|
2	SAMPLE/BLANK/STANDARDS MONTH ANALYZED	MM
1	Delimiter	|
2	SAMPLE/BLANK/STANDARDS DAY ANALYZED	DD
1	Delimiter	|
2	SAMPLE/BLANK/STANDARDS HOUR ANALYZED	HH
1	Delimiter	|
2	SAMPLE/BLANK/STANDARDS MINUTE ANALYZED	MM
2	Delimiters	||
2	SAMPLE VOL UNITS	"ML"/blank[7]
1	Delimiter	|
5	SAMPLE VOL	Numeric[8]

[5]"0" if not applicable (calibration, tune, etc.); "1" for water.

[6]"RIN" for reinjection; "REX" for re-extractions; "REJ" for rejected samples; "SRN" for dilutions; and leave blank (empty field with zero length) when none of the previous conditions apply. In case of multiple operations on a sample, the final operation will be indicated (e.g., reinjection of a dilution; AAA12DLRE would have a QC Code of "RIN").

[7]Sample VOL unit is ML (milliliters) for liquids. Leave blank (zero length) if not applicable.

[8]Sample VOL is the volume in milliliters for liquid. Sample VOL includes the purge volume.

Sample Header Data Record (Type 20) (Con't)

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
1	Delimiter	|
3	ANALYTE COUNT	Numeric[9]
3	Delimiters	|||
5	RECORD SEQUENCE NO.	Numeric
4	CHECKSUM	Character

[9]Counts TCL analytes, surrogates, deuterated monitoring compounds (DMC), internal standards, and all peaks reported for multi-component PCBs. Do not include the count for TICs in this field. For calibrations, also count DFTPP, if included in calibration solution.

9.4 Sample Header Data Record (Type 21)

Use: Continuation of Type 20.
Position: Follows the Type 20 to which it applies.

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"21"
1	Delimiter	|
1	PURGE	"N" (for VOA); blank (for SV or PEST[10]
3	Delimiters	|||
1	EXTRACTION	S/C/H/blank (for all volatile samples)[11]
2	Delimiters	||
6	CLIENT NUMBER	Character
1	Delimiter	|
14	LAB FILE/SAMPLE ID	Character[12]
1	Delimiter	|
4	YEAR EXTRACTED	YYYY/blank (for volatiles)
1	Delimiter	|
2	MONTH EXTRACTED	MM/blank (for volatiles)
1	Delimiter	|
2	DAY EXTRACTED	DD/blank (for volatiles)
2	Delimiters	||
4	YEAR RECEIVED	YYYY/blank (for standards, tunes, and blanks)
1	Delimiter	|
2	MONTH RECEIVED	MM/blank (for standards, tunes, and blanks)
1	Delimiter	|
2	DAY RECEIVED	DD/blank (for standards, tunes, and blanks)
1	Delimiter	|
7	IDENTIFICATION/LOT NUMBER OF LCS	Character[13]
1	Delimiter	|

[10]"N" for not heated purge. All low concentration volatile samples are required to be purged at ambient temperature.

[11]"S" for separatory funnel; "C" for continuous liq-liq without hydrophobic membrane; "H" for continuous liq-liq with hydrophobic membrane; blank (zero length field) for all volatile samples.

[12]Lab File ID for volatile and semivolatile analyses. Lab Sample ID for pesticides in same format as on forms.

[13]If the LCS solution was purchased by the Contractor from a third party, report the identification number used by the third party to identify the LCS lot, if available (Form 3). If the LCS solution was prepared in-house, leave this entry blank.

Sample Header Data Record (Type 21) (Con't)

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
8	INJECTION VOLUME	Numeric/blank (for VOA)[14]
2	Delimiters	||
5	RECORD SEQUENCE NO.	Numeric
4	CHECKSUM	Character

[14]Injection volume, in µL, for SVs and PESTs.

9.5 Sample Condition Record (Type 22)

Use: Continuation of type 20. Used to describe additional Sample Conditions.
Position: Follows the type 20 and 21 to which it applies.

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"22"
1	Delimiter	|
4	CALIBRATION YEAR	YYYY/blank (for PEST)[15]
1	Delimiter	|
2	CALIBRATION MONTH	MM/blank (for PEST)
1	Delimiter	|
2	CALIBRATION DAY	DD/blank (for PEST)
1	Delimiter	|
2	CALIBRATION HOUR	HH/blank (for PEST)
1	Delimiter	|
2	CALIBRATION MINUTE	MM/blank (for PEST)
1	Delimiter	|
14	CALIBRATION FILE ID	Character/blank (for PEST)[16]
4	Delimiters	||||
8	EXTRACT VOLUME	Numeric/blank (for VOA)[17]
1	Delimiter	|
8	DILUTION FACTOR	Numeric[18]
3	Delimiters	|||
5	LEVEL	Numeric/blank (for VOA/SV)[19]
1	Delimiter	|
5	RECORD SEQUENCE NO.	Numeric
4	CHECKSUM	Character

[15]For volatiles and semivolatiles, enter the date and time of analysis of the most recent 5 ug/L (VOAs) or the 20 ng/uL (SVs) standard run prior to the sample reported in the associated type 20 record. Leave blank for pesticides.

[16]Lab File ID of standard specified in 15 above (volatiles/semivolatiles only). This field must match the Lab File ID on Type 21 for the associated calibration (VSTD005/SSTD020). Leave blank for pesticides.

[17]Enter the Concentrated Extract Volume for all SV and PEST. The value should be reported in microliters.

[18]Dilution factor of sample analyzed.

[19]Concentration level of Pesticide Individual Mix A and B standards. Concentration of low point, mid point and high point calibration standards as a multiplier of low point. Low point = 1.0; Mid point = 4.0; High point $ 16.0.

9.6 Associated Injection and Counter Record (Type 23)

Use: Continuation of type 20. Used to identify associated blanks and tunes, and the number of surrogates/DMCs and spikes outside of the Quality Control (QC) limits and the number of tentatively identified compounds (TICs).

Position: Follows the type 20, 21, and 22 to which it applies.

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"23"
1	Delimiter	|
1	INSTRUMENT PERFORMANCE CHECK (IPC/TUNE) LABEL	"P" (for BFB and DFTPP IPC) or blank (for pesticides)
1	Delimiter	|
4	IPC/TUNE INJECTION YEAR	YYYY/blank (for PEST)
1	Delimiter	|
2	IPC/TUNE INJECTION MONTH	MM/blank (for PEST)
1	Delimiter	|
2	IPC/TUNE INJECTION DAY	DD/blank (for PEST)
1	Delimiter	|
2	IPC/TUNE INJECTION HOUR	HH/blank (for PEST)
1	Delimiter	|
2	IPC/TUNE INJECTION MINUTE	MM/blank (for PEST)
1	Delimiter	|
14	DFTPP/BFB LAB FILE ID	Character/blank (for PEST)
1	Delimiter	|
2	VOLATILE STORAGE BLANK LABEL	"HB" (for VOA) or blank (for SV and PEST)
1	Delimiter	|
4	STORAGE BLANK INJECTION YEAR	YYYY/blank (for SV and PEST)
1	Delimiter	|
2	STORAGE BLANK INJECTION MONTH	MM/blank (for SV and PEST)
1	Delimiter	|
2	STORAGE BLANK INJECTION DAY	DD/blank (for SV and PEST)
1	Delimiter	|
2	STORAGE BLANK INJECTION HOUR	HH/blank (for SV and PEST)
1	Delimiter	|
2	STORAGE BLANK INJECTION MINUTE	MM/blank (for SV and PEST)
1	Delimiter	|
14	STORAGE BLANK LAB FILE ID (VOA ONLY)	Character
4	Delimiters	||||

Associated Injection and Counter Record (Type 23) (Con't)

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	METHOD BLANK LABEL	"MB"/blank (for standard, tune, and method blanks)
1	Delimiter	|
4	METHOD BLANK INJECTION YEAR	YYYY/blank (for standard, tune, and method blanks)
1	Delimiter	|
2	METHOD BLANK INJECTION MONTH	MM/blank (for standard, tune, and method blanks)
1	Delimiter	|
2	METHOD BLANK INJECTION DAY	DD/blank (for standard, tune, and method blanks)
1	Delimiter	|
2	METHOD BLANK INJECTION HOUR	HH/blank (for standard, tune, and method blanks)
1	Delimiter	|
2	METHOD BLANK INJECTION MINUTES	MM/blank (for standard, tune, and method blanks)
1	Delimiter	|
14	METHOD BLANK LAB FILE (for VOA and SV)/SAMPLE ID (for PEST)	CHARACTER
1	Delimiter	|
1	SURROGATE (for PEST)/ DMC (for VOA and SV) RECOVERY LABEL	"P" for % recoveries/blank (for STD/IPC)
1	Delimiter	|
2	SURROGATE (for PEST)/ DMC (for VOA and SV) RECOVERIES OUT	Numeric[20]
1	Delimiter	|
1	TIC LABEL	"T" (for VOA and SV TICs)/blank (for PEST)
1	Delimiter	|
2	NO. OF TICS	Numeric
1	Delimiter	|
1	SPIKE RECOVERY LABEL	"S" for LCS (Pest)/MS/MSD (all fractions)/blank for anything else
1	Delimiter	|

[20]This will be the number of surrogate (for PEST) or DMC (for VOA and SV) recoveries outside QC limits for a specific column. It should not be cumulative of the two columns for pesticides.

Associated Injection and Counter Record (Type 23) (Con't)

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	SPIKE RECOVERIES OUT	Numeric/blank[21]
1	Delimiter	|
1	RPD LABEL	"R" for RPD/blank[22]
1	Delimiter	|
2	RPD OUT	Numeric
1	Delimiter	|
5	RECORD SEQUENCE NO.	Numeric
4	CHECKSUM	Character

[21]Enter the number of spike recoveries out. Enter "0"(zero) if none of the spike recoveries are outside of the QC limit.

[22]"R" for Matrix Spike/Matrix Spike Duplicate Recovery Relative Percent Difference. Leave blank for all other samples.

9.7 Sample Cleanup Record (Type 27)

Use: Continuation of type 20. Used to identify sample/blank cleanup procedures and QC results.
Position: Follows type 20, 21, 22, and 23 to which it applies.

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"27"
8	Delimiters	||||||||
1	FLORISIL CLEANUP TYPE	"F"/blank (for VOA and SV)
1	Delimiter	|
4	FLORISIL LOT CHECK YEAR	YYYY/blank (for VOA and SV)
1	Delimiter	|
2	FLORISIL LOT CHECK MONTH	MM/blank (for VOA and SV)
1	Delimiter	|
2	FLORISIL LOT CHECK DAY	DD/blank (for VOA and SV)
1	Delimiter	|
2	FLORISIL LOT CHECK HOUR	HH/blank (for VOA and SV)
1	Delimiter	|
2	FLORISIL LOT CHECK MINUTE	MM/blank (for VOA and SV)
1	Delimiter	|
14	FLORISIL DATA DESCRIPTOR	Character[23]
1	Delimiter	|
1	SULFUR CLEANUP	Y/N (for PEST)/blank (for VOA and SV)
1	Delimiter	|
2	SULFUR BLANK LABEL	"SB"/blank (if no separate sulfur blank was prepared for pesticides; also blank for VOA and SV)
1	Delimiter	|
4	SULFUR BLANK INJECTION YEAR	YYYY/blank (for VOA and SV)
1	Delimiter	|
2	SULFUR BLANK INJECTION MONTH	MM/blank (for VOA and SV)
2	Delimiters	||
2	SULFUR BLANK INJECTION DAY	DD/blank (for VOA and SV)
1	Delimiter	|
2	SULFUR BLANK INJECTION HOUR	HH/blank (for VOA and SV)
1	Delimiter	|
2	SULFUR BLANK INJECTION MINUTE	MM/blank (for VOA and SV)
1	Delimiter	|
14	SULFUR BLANK LABORATORY/ SAMPLE ID	Character
1	Delimiter	|
5	RECORD SEQUENCE NO.	Numeric
4	CHECKSUM	Character

[23]Lab Sample ID of associate Florisil lot check. This is a unique identifier assigned to a lot of Florisil cartridges.

9.8 Results Data Record (Type 30)

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"30"
1	Delimiter	|
1	ANALYTE LABEL	"C" for CAS Number (blank for unknown TICs)
1	Delimiter	|
9	CAS NUMBER	Numeric (for TCL, surrogates, DFTPP, BFB, DMC, internal standards, and identified TICs)
1	Delimiter	|
9	INTERNAL STD. CAS NUMBER	Numeric
1	Delimiter	|
5	CONCENTRATION UNITS	Character "UG/L" or "NG" (for amount added)
1	Delimiter	|
3	RESULT QUALIFIER	Character[24,25]
1	Delimiter	|
13	RESULTS	Numeric[26]
1	Delimiter	|
5	FLAGS	Character[27]
1	Delimiter	|
1	AMOUNT ADDED LABEL	"A" for Amt. added[28]
1	Delimiter	|
13	AMOUNT ADDED	Numeric
1	Delimiter	|

[24]When a Type 20 Record is used for calibration summary (MNC), the associated Type 30 Record uses "AVG" for average RRFs and Mean Calibration Factors. See Exhibit H Section 10.1.2.

[25]For pesticide sample analysis, if an analyte is detected in only one of the two column analyses, report the analyte as "not detected" in both runs. Report result qualifier, for each column, as BDL. See Section 10.3.2 for result qualifiers.

[26]Leave this field blank only when reporting non-detects.

[27]A maximum of five flags (D,E,J,B,A,P,X,Y,Z, or N) with no space between the flags can be reported, each representing a qualification of the result as described in Exhibit B. For surrogates and DMC (in SV), the "D" flag will indicate surrogates diluted out.

[28]For Matrix Spike/Matrix Spike Duplicate (MS/MSD) analysis (for VOA, SV, and PEST), Laboratory Control Sample analysis and surrogate (for PEST), DMC (for VOA and SV). Nominal Amount for Pesticides (Forms 7LCG/7LCH). Spike added for Florisil (Form 9LCA).

Results Data Record (Type 30) (Con't)

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
1	CRQL LABEL	"U" for "undetected" or blank when analyte is detected
1	Delimiter	|
13	CRQL	Numeric
1	Delimiter	|
1	RSD LABEL	"R" for % Resolution/RSD[29]
1	Delimiter	|
5	RSD VALUE	Numeric
1	Delimiter	|
1	LCS MS/MSD REC LABEL	"P" for % recovery [LCS, MS/MSD]/blank (for all other samples, standards, tunes, blanks, calibrations)
1	Delimiter	|
5	% RECOVERY	Numeric/blank (for all other samples except LCS, MS/MSD)
2	Delimiters	||
1	RPD LABEL	"D" for pesticide calibration verification (%D)/blank for everything else
1	Delimiter	|
5	RPD VALUE	Numeric/blank[30]
1	Delimiter	|
1	DMC/SURR/SPIKE RECOVERY LABEL	"S" for % recovery/blank (for non-surrogate/and non-spike analytes non-DMC
1	Delimiter	|
5	DMC/SURR/SPIKE RECOVERY	% Recovery/blank[31]

[29]"R" for % Resolution (Forms 6LCJ, 6LCK, 6LCL, and 6LCM) or for RSD of Response factors under Calibration summary (MNC) Type 20. (Blank for VOA and SV fractions.)

[30]RPD for MS/MSD recoveries, or %D for pesticides Calibration Verification (Form 7LCG/7LCH). Otherwise, leave blank.

[31]Surrogate (for PEST)/DMC (for VOA and SV) or Spike (Forms 2, Form 9LCA) recovery. Leave blank for non-spike analytes.

Results Data Record (Type 30) (Con't)

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
1	Delimiter	|
1	MEAN CONCENTRATION LABEL	"M" for Mean conc. (for multicomponent PEST only)/blank (for VOA and SV)
1	Delimiter	|
13	MEAN CONCENTRATION	Numeric (for PEST)/blank (for VOA and SV)[32]
1	Delimiter	|
1	PERCENT DIFFERENCE LABEL	"F" or "P" (PEST)/blank (for VOA and SV field sample analysis)[33]
1	Delimiter	|
5	PERCENT DIFFERENCE	Numeric
1	Delimiter	|
1	INTERNAL STANDARD AREA LABEL	"I" for IS Area (for VOA and SV)/blank (for PEST)
1	Delimiter	|
13	INTERNAL STANDARD AREA	Numeric (for VOA and SV)/blank (for PEST)
1	Delimiter	|
5	RECORD SEQUENCE NO.	Numeric
4	CHECKSUM	Character

[32]Mean Concentration for Multicomponent analytes detected in pesticide analyses.

[33]"P" for Percent Difference between concentrations from two columns in pesticide analyses, or "F" for Percent Difference between average RRF (initial calibration) and RRF5/RRF20 (continuing calibration) in VOA/SV analyses. Leave blank for volatile and semivolatile sample, blank, and tune analysis.

9.9 Auxiliary Data Record (Type 32)

Use: Used to report retention time (in minutes) for Internal Standards and for TICs (for Volatiles and Semivolatiles). Used to report retention time data and percent breakdown (for pesticides).

Position: Follows type 30. (Record will only be required as specified above.)

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"32"
3	Delimiters	|||
2	RETENTION TIME LABEL	"RT"
1	Delimiter	|
5	RETENTION TIME	Numeric
1	Delimiter	|
3	FIRST LIMIT LABEL	"RTF"
1	Delimiter	|
5	RT WINDOW LOWER LIMIT	Numeric
1	Delimiter	|
3	SECOND LIMIT LABEL	"RTT"
1	Delimiter	|
5	RT WINDOW UPPER LIMIT	Numeric
2	Delimiters	||
2	% BREAKDOWN LABEL	"PB" for % breakdown/blank (for VOA and SV)
1	Delimiter	|
5	% BREAKDOWN	Numeric (DDT/ENDRIN)/blank (for VOA and SV)
1	Delimiter	|
5	COMBINED % BREAKDOWN	Numeric/blank (for VOA and SV)[34]
2	Delimiters	||
1	PEAK	1 THROUGH 5 (for pesticide multicomponent compounds)/blank (for VOA and SV)[35]
1	Delimiter	|
5	RECORD SEQUENCE NO.	Numeric
4	CHECKSUM	Character

[34]The combined %breakdown will be reported on both the record type 32s for DDT and Endrin.

[35]For positively identified compounds, a minimum of 3 peaks and a maximum of 5 peaks are allowed. Types 30 and 32 will be repeated for each peak that is reported (a minimum of three, a maximum of five times). This is for multicomponent analytes in pesticide analyses.

9.10 Name Record (Type 33)

Use: This record type is used for volatile and semivolatile analyses only to carry an analyte name for TICs. This record is not used for pesticide analysis.
Position: Follows types 30 and 32 for TICs.

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"33"
1	Delimiter	|
67	NAME OF COMPOUND	Character
1	Delimiter	|
5	RECORD SEQUENCE NO.	Numeric
4	CHECKSUM	Character

9.11 Instrumental Data Readout Record (Type 36)

Use: This record type is only used for volatile and semivolatile analyses to describe decafluorotriphenylphosphine/4-bromofluorobenzene (DFTPP/BFB) percent abundances. This record is not used for pesticide analysis.

Position: Follows type 30 for DFTPP/BFB data.

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"36"
1	Delimiter	|
1	MASS LABEL	"M"
3	Delimiters	|||
3	FIRST MASS (DFTPP/BFB)	Numeric (DFTPP for SV or BFB for VOA)
2	Delimiters	||
5	FIRST PERCENT RELATIVE ABUNDANCE	Numeric
1	Delimiter	|
3	SECOND MASS	Numeric
1	Delimiter	|
5	SECOND PERCENT RELATIVE ABUNDANCE	Numeric
1	Delimiter	|
5	PERCENT MASS OF 69	Numeric, DFTPP only/blank (for VOA)
1	Delimiter	|
3	THIRD MASS	Numeric
1	Delimiter	|
5	THIRD PERCENT RELATIVE ABUNDANCE	Numeric
2	Delimiters	||
3	FOURTH MASS	Numeric
1	Delimiter	|
5	FOURTH PERCENT RELATIVE ABUNDANCE	Numeric
1	Delimiter	|
5	PERCENT MASS OF 69	Numeric, DFTPP only/blank (for VOA)
1	Delimiter	|
3	FIFTH MASS	Numeric
1	Delimiter	|
5	FIFTH PERCENT RELATIVE ABUNDANCE	Numeric
1	Delimiter	|
5	PERCENT MASS OF 174	Numeric, BFB only/blank (for SV)
1	Delimiter	|
3	SIXTH MASS	Numeric
1	Delimiter	|

Instrumental Data Readout Record (Type 36) (Con't)

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
5	SIXTH PERCENT RELATIVE ABUNDANCE	Numeric
2	Delimiters	||
3	SEVENTH MASS	Numeric
1	Delimiter	|
5	SEVENTH PERCENT RELATIVE ABUNDANCE	Numeric
1	Delimiter	|
5	PERCENT MASS OF 174	Numeric, BFB only/blank (for SV)
1	Delimiter	|
3	EIGHTH MASS	Numeric
1	Delimiter	|
5	EIGHTH PERCENT RELATIVE ABUNDANCE	Numeric
1	Delimiter	|
5	PERCENT MASS OF 174	Numeric, BFB only/blank (for SV)
1	Delimiter	|
3	NINTH MASS	Numeric
1	Delimiter	|
5	NINTH PERCENT RELATIVE ABUNDANCE	Numeric
1	Delimiter	|
5	PERCENT MASS OF 176	Numeric, BFB only/blank (for SV)
1	Delimiter	|
3	TENTH MASS	Numeric/blank (for VOA)
1	Delimiter	|
5	TENTH PERCENT RELATIVE ABUNDANCE	Numeric/blank (for VOA)
2	Delimiters	||
3	ELEVENTH MASS	Numeric/blank (for VOA)
1	Delimiter	|
5	ELEVENTH PERCENT RELATIVE ABUNDANCE	Numeric/blank (for VOA)
2	Delimiters	||
3	TWELFTH MASS	Numeric/blank (for VOA)
1	Delimiter	|
5	TWELFTH PERCENT RELATIVE ABUNDANCE	Numeric/blank (for VOA)
2	Delimiters	||
3	THIRTEENTH MASS	Numeric/blank (for VOA)
2	Delimiters	||
5	THIRTEENTH PERCENT RELATIVE ABUNDANCE	Numeric/blank (for VOA)
1	Delimiter	|
5	PERCENT MASS OF 442	Numeric, DFTPP only (blank for VOA)
1	Delimiter	|
5	RECORD SEQUENCE NO.	Numeric
4	CHECKSUM	Character

9.12 Comment Record (Type 90)

Use: To provide for operator-entered comments.
Position: May occur anywhere in the file after the type 10 record.

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"90"
1	Delimiter	|
67	ANY COMMENT	Character
1	Delimiter	|
5	RECORD SEQUENCE NO.	Numeric
4	CHECKSUM	Character

10.0 DEFINITIONS OF VARIOUS CODES USED IN AGENCY STANDARD RECORDS

10.1 Quality Control and Related Codes (QCC) in Type 20 Records

10.1.1 NOTE: These codes appear in the quality control (QC) code fields of type 20 records. They are used to indicate the type of data that is being reported.

QCC	Name	Definition
LRB	LABORATORY (REAGENT) BLANK	The "Method Blank" (Exhibit G).
LIB	LABORATORY INSTRUMENT BLANK	The "Instrument Blank".
LSB	LABORATORY SULFUR BLANK	If different from "Method Blank" (pesticides).
LHB	LABORATORY STORAGE BLANK	The storage blank (volatiles).
FRB	FIELD BLANK	This is any sample that is submitted from the field and is identified as a blank. This includes trip blanks, rinsates, equipment blanks, etc.
FRM	FIELD REFERENCE SAMPLE	This is any sample that is submitted for a Case and is identified as a Performance Evaluation (PE) sample.
LSD	LABORATORY SPIKE DUPLICATE BACKGROUND (ORIGINAL) VALUES	An environmental sample which is analyzed according to the analytical method, and subsequently used for the matrix spike, and the matrix spike duplicate (Exhibit G).
LF1	LABORATORY SPIKE SAMPLE _FINAL_ FIRST MEMBER	The “Matrix spike” (Exhibit G); must precede LF2.
LF2	LABORATORY SPIKE SAMPLE _FINAL_ SECOND MEMBER	The “Matrix spike Duplicate” (Exhibit G).
LCM	LABORATORY CONTROL SAMPLE	The Laboratory Control Sample (LCS) (Exhibit G).
LPC	LABORATORY PERFORMANCE CHECK SOLUTION	A solution of DFTPP (SV) or BFB (VOA) or method analytes (PEST/PCB) used to evaluate the performance of an instrument with respect to a defined set of criteria (Tune or Resolution Check Sample) (Exhibit G).
FLO	FLORISIL CHECK SOLUTION	A solution of pesticides used to check recovery from each lot of Florisil cartridges. These recovery results will be provided in every production run where associated samples are analyzed.

QCC	Name	Definition
CLM	INITIAL CALIBRATION - MULTI-POINT	The Initial Calibration for GC/MS (Exhibit G), or the Initial Individual Standard Mixes (A, B) for pesticides (Exhibit D PEST). Response factors (GC/MS) or Calibration Factors (pesticides) will be reported on the following type 30 records.
CLS	INITIAL CALIBRATION SINGLE POINT	The Initial Toxaphene/Aroclor Mixes used to determine all calibration factors (Exhibit D PEST).
CLC	CONTINUING CALIBRATION CHECK	The continuing calibration (VSTD005/ SSTD020) for GC/MS.
CLE	CONTINUING PERFORMANCE CHECK	The subsequent Individual Standard Mixes (A,B), Performance Evaluation Mixture, and for subsequent injections of Toxaphene/Aroclor mixes for pesticides (Exhibit D PEST).
CLD	DUAL PURPOSE CALIBRATION	A calibration solution as above used both as an initial calibration (CLM) and a continuing check (CLC).

10.1.2 The following QCC values are used on type 20 records which act as a header, and indicate that additional (usually calculated) analyte specific data will be present on type 30 (and following type) records. Usually, these data will apply to an entire production run, in which case they will appear immediately following the type 10 record or type 11 record if present. If the data apply to only a portion of the samples in the run, they shall be placed immediately preceding the samples to which they apply. Much of the rest of the information in the type 20 record may be blank, indicating that these data do not apply to these results.

MNC	MEAN VALUES FROM CALIBRATIONS	The data following represent mean values and percent RSDs from the initial calibration (GC/MS) or the mean calibration factors, mean retention times, and retention time windows (pesticides).

10.2 Codes For Sample Medium (Matrix, Sources)

Medium	Code
All Media, Specific Medium not Applicable. Use for Calibrations, Tunes, etc.	0 (zero)
Water	1

10.3 List of Sample and Result Qualifiers

Definition: A sample qualifier consists of three characters which act as an indicator of the fact and the reason that the subject analysis (a) did not produce a numeric result, or (b) produced a numeric result for an entire sample but it is qualified in some respect relating to the type or validity of the result.

10.3.1 Sample Qualifiers

Qualifier	Full Name	Definition
RIN	RE-ANALYZED	The indicated analysis results were generated from a re-injection of the same sample extract or aliquot (RE SUFFIX).
REX	RE-PREPARED	The indicated analysis results were generated from a re-extraction of the same sample (RE SUFFIX).
REJ	REJECTED	The results for the entire sample analysis have been rejected for an unspecified reason by the laboratory. For initial calibration data, these data were not utilized in the calculation of the mean.
SRN	DILUTED	The indicated analysis results were generated from a dilution of the same sample (DL SUFFIX).

10.3.2 Result Qualifiers in Type 30 Records

A result qualifier consists of three characters which act as an indicator of the fact and the reason that the subject analysis (a) did not produce a numeric result, or (b) produced a numeric result for a single analyte but it is qualified in some respect relating to the type or validity of the result. This qualifier is complementary to the flags field on a type 30 record. A tentatively identified compound (TIC) **must** have either a TIE, TFB, or PRE result qualifier.

BDL	BELOW DETECTABLE LIMITS	Indicates compound was analyzed for but not detected (Form 1 "U" Flag).
NAR	NO ANALYSIS RESULT	There is no analysis result required for this subject parameter.
AVG	AVERAGE VALUE	Average value -- used to report a range of values (e.g., relative response factors).
CBC	CANNOT BE CALCULATED	The analysis result cannot be calculated because an operand value is qualified (e.g., identifies analytes whose internal standard is not found) (Form 1 "X" Flag).

LTL	LESS THAN LOWER CALIBRATION LIMIT	Analysis result is from a diluted sample (DL suffix) and may be less accurate than the result from an undiluted sample (Form 1 "D" Flag).
GTL	GREATER THAN UPPER CALIBRATION LIMIT	Actual value is known to be greater than the upper calibration range (Form 1 "E" Flag).
LLS	LESS THAN LOWER STANDARD	The analysis result is less than the sample quantitation limit (Form 1 "J" Flag).
TIE	TENTATIVELY IDENTIFIED ESTIMATED VALUE	The indicated analyte is a tentatively identified analyte; its concentration has been estimated (Form 1LCF or 1LCG "J" Flag).
REJ	REJECTED	Results for the analyte are rejected by the laboratory.
STD	INTERNAL STANDARD	The indicated compound is an internal standard.
STB	INTERNAL STANDARD BELOW DETECTION LIMITS	A combination of "STD" and "BDL".
FBK	FOUND IN BLANK	The indicated compound was found in the associated method blank (LRB) as well as the sample (Form 1 "B" Flag).
TFB	TENTATIVELY IDENTIFIED AND FOUND IN BLANK	A Combination of "TIE" and "FBK" (Form 1LCF or 1LCG "B" Flag).
NRP	NON-REPRODUCIBLE	Results of two or more injections are not comparable (Form 1LCE "P" flag), e.g., Aroclor target analyte with greater than 25% difference between mean concentrations of the two column analyses.
PRE	PRESUMPTIVE PRESENCE	Presumptive evidence of presence of material for TIC (Form 1LCF or 1LCG "N" Flag).
ALC	ALDOL CONDENSATION	Labels a suspected Aldol Condensation product for TICs (Form 1LCG)